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(71)Applicant : TDK CORP
(72)Inventor : FUKUNO AKIRA
NAKAMURA HIDEKI
NISHIZAWA KOICHI

(54) SINTERED MAGNET AND ITS PRODUCTION

(57)Abstract:
PURPOSE: To provide an inexpensive thin magnet by suppressing fluctuation in the dimensions of an R-T-B based sintered magnet at the time of sintering thereby eliminating the need of grinding after sintering.
CONSTITUTION: A molded item of a powder containing R (at least one kind of rare earth element containing Y), T (Fe and/or Co) and B where the average particle size of main phase comprising $R_2T_{14}B$ is 20μm or above, and a powder containing 75-97wt.% of R and the remainder of Fe and/or Co which is blocked by a screen of 38μm mesh or above but passes a screen of 500μm mesh or less is sintered to produce a sintered magnet containing 3-15vol.% of closed pores.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]
[0001]
[Industrial Application] This invention relates to the rare earth sintered magnet with small contraction and its manufacture approach at the time of sintering.
[0002]
[Description of the Prior Art] As a rare earth magnet which has high performance, the thing of energy product 32MGOe is mass-produced with the Sm-Co system magnet by powder-metallurgy processing. Moreover, R-T-B system magnets (T is Fe, or Fe and Co), such as a Nd-Fe-B magnet, are developed in recent years, and the sintered magnet is indicated by JP,59-46008,A. A R-T-B system magnet has a cheap raw material compared with a Sm-Co system magnet. The powder metallurgy process (dissolution -> casting -> ingot coarse-grinding -> pulverizing -> shaping -> sintering -> magnet) of the conventional Sm-Co system is applicable to manufacture of a R-T-B system sintered magnet.
[0003] With the R-T-B system magnet, the BONDIDDO magnet which combined the magnet powder other than a sintered magnet with the resin binder metallurgy group binder is also put in practical use. Since the dimension in the case of shaping is maintained mostly, a BONDIDDO magnet has high dimensional accuracy and does not need configuration processing after manufacture. However, since the polycrystal particle which consists of a fine crystal manufactured with quenching methods, such as the single rolling method, is used for the BONDIDDO magnet of the R-T-B system industrialized, anisotropy-izing by shaping among a magnetic field etc. is difficult. Since coercive force is decreasing sharply by distortion, oxidization, etc. by grinding, the pulverized powder of a R-T-B system sintered magnet cannot be used as raw material powder of a BONDIDDO magnet. In addition, the pulverized powder of a R-T-B system alloy ingot is made to react with hydrogen, it decomposes into a rare earth hydride, the way ghost of T, and T, and the proposal which deposits the fine crystal to which crystal orientation was equal within each particle is also made by carrying out a dehydrogenation at predetermined temperature. Although magnetic field orientation is possible for the polycrystal particle obtained by this approach and high coercive force is acquired with a fine crystal, since a process becomes complicated in order to use hydrogen, it is not put in practical use.
[0004] A high property is acquired in order to obtain an anisotropic magnet easily on the other hand in order to fabricate the powder which consists of a single crystal particle substantially all over a magnetic field in a R-T-B system sintered magnet, and not to use a binder moreover. However, in a sintering process, a Plastic solid contracts remarkably to sintering reaction time, and since the contraction is uneven, maintenance of the dimensional accuracy of a Plastic solid is difficult. This contraction changes with the amount of preferred orientation of the particle in a Plastic solid, dispersion of a consistency, etc. a direction vertical to the direction of an easy axis, and it in an anisotropy sintered magnet -- contraction -- differing -- for example, the consistency of a Plastic solid -- 4.3 g/cm3 it is -- the time -- a direction vertical to about 22% and it in the direction of an easy axis -- about 15% -- becoming -- the consistency after sintering -- 7.55 g/cm3 It reaches.
[0005] In the case of the thing of thin meat, such a dimensional change especially in an anisotropy sintered magnet becomes with a problem with a ring-like magnet or a tabular magnet. It is because curvature will occur if contraction becomes an ununiformity in a light-gage magnet. Then, on the

occasion of commercial production, in order to amend such a dimensional change, the grinding process of the sintered compact is carried out. However, there is a problem which is described below in a grinding process.

[0006] ** The amount of ingredient loss of the sintered compact at the time of a grinding process becomes large. For example, since it is necessary to manufacture a sintered compact with a thickness of about 3mm, and to carry out grinding of the vertical side of this first when 1mm curvature occurs, in case a light-gage tabular magnet with a thickness of 1mm is produced, two thirds of ingredients is lost. In order to avoid such loss, even when quarrying out two or more light-gage tabular magnets in thickness of 1mm from one heavy-gage base material, about 40% of loss will arise that the face width of the cutter for grinding is 0.6mm. Moreover, since the mechanical strength is small, a chip and a crack will tend to generate the sintered compact of thin meat in the case of the impact at the time of processing, or handling, and the yield will become low.

[0007] ** Magnetic properties fall. Depending for the coercive force of a Nd2 Fe14B system sintered magnet on existence of Nd rich phase of a grain boundary is reported in detail in various papers etc. In case the sintered magnet of this system is processed, a crack etc. will arise in the grain boundary of the field near a processing side with stress, and coercive force will be lost in the field from a processing side to a depth of 0.1-0.2mm. Even if disappearance of a magnet property [/ near the processing side] can ignore with a heavy-gage magnet, with a light-gage magnet, effect will be large and magnetic-properties degradation as the whole magnet will become clear. In addition, it is possible to remove the field where coercive force disappeared by processing by acid etching, and the amount of loss of a sintered compact will increase further, and a manufacturing cost will also increase.

[0008] With the light-gage anisotropic magnet with which longitudinal direction die length / thickness amounts to ten or more, the Sm-Co system BONDIDDO magnet is used and the cost high usually poses a problem from such a situation. Although the light-gage sintered magnet of a R-T-B system also exists, processing for size adjustment is indispensable, and since the ingredient yield in the case of processing moreover becomes 20 - 30%, it has the cost high too.

[0009]

[Problem(s) to be Solved by the Invention] By suppressing the dimensional change at the time of sintering in manufacture of a R-T-B system sintered magnet, this invention makes the grinding process after sintering unnecessary, and aims at offering a cheap light-gage magnet.

[0010]

[Means for Solving the Problem] Such an object is attained by this invention of following the (1) - (15).

(1) The sintered magnet which is a sintered magnet containing R (R is at least one sort of the rare earth elements containing Y), T (T is Fe, or Fe and Co), and B, and is characterized by 3-15 volume % Including a close hole.

(2) Consistencies are 7.15 g/cm3. Sintered magnet of the above (1) which is the following.

(3) the average projection cross section per close hole -- 1000-30000 micrometers 2 it is -- the above (1) or sintered magnet of (2).

(4) One sintered magnet of above-mentioned (1) - (3) whose ratios of an open hole are below 2 volume %.

(5) R -- 30 - 45 -- % of the weight -- B -- 0.5 - 3.5 -- % of the weight -- containing -- the remainder -- substantial -- T -- it is -- the above -- (-- one --) - (-- four --) -- either -- a sintered magnet .

(6) R (R is at least one sort of the rare earth elements containing Y), T (T) The sintered magnet which contains B and it is Fe, or Fe and Co After fabricating the mixture of the powder of the hardener for the main phases, and the powder of the hardener for grain boundary phases, Are the approach of manufacturing by sintering and said hardener for the main phases has the crystal grain which consists of R2 T14B substantially. Mean particle diameter is 20 micrometers. Are above and said hardener for grain boundary phases contains R 70 to 97% of the weight. The remainders are Fe and/or Co substantially and an aperture is 38 micrometers. It remains to the above sieve and an aperture is 500 micrometers. The manufacture approach of the sintered magnet characterized by being what passes the following sieves.

(7) The manufacture approach of the sintered magnet the above (6) which makes the ratio of the

powder of the hardener for grain boundary phases in said mixture 2 - 20 % of the weight.
(8) The manufacture approach of of the above (6) or the sintered magnet of (7) with which Nd occupies 50% or more of R of said hardener for grain boundary phases.
(9) The manufacture approach of one sintered magnet of above-mentioned (6) - (8) which manufactures said hardener for grain boundary phases with a melt quenching method.
(10) The manufacture approach of one sintered magnet of above-mentioned (6) - (9) which sinters at the temperature more than the melting point of said hardener for grain boundary phases.
(11) The manufacture approach of one sintered magnet of above-mentioned (6) - (10) which sinters in a vacuum.
(12) Consistency 5.5 g/cm3 Consistency change is the above Plastic solid 0.2 g/cm3 The manufacture approach of one sintered magnet of above-mentioned (6) - (11) which has the process sintered so that it may become the above.
(13) Anti-chip box reinforcement is 2 0.3 kgf(s)/mm. The manufacture approach of one sintered magnet of above-mentioned (6) - (12) which sinters the Plastic solid which it is above.
(14) Compacting pressure is 8 t/cm2. The above (6) which it is above The manufacture approach of one sintered magnet of - (13).
(15) The manufacture approach of one sintered magnet of above-mentioned (1) above-mentioned [which manufactures one sintered magnet of - (5)] (6) - (14).
[0011]
[Function and Effect] It is about 55% of consistency (about 4.2 g/cm3) of the consistency (theoretical density: about 7.6 g/cm3) when assuming that the conventional Plastic solid for Nd2 Fe14B sintered magnets does not have a hole, and about 45% of hole is included. And since eburnation is carried out to about 99% of theoretical density by sintering, the rate of a volumetric shrinkage will become large.
[0012] On the other hand, in this invention, contraction is small suppressed by forming a close hole by the predetermined ratio in a magnet in the case of sintering. Since the close hole is not open for free passage to the magnet exterior, unlike the open pore (open hole) of the conventional partial-loss-by-fire join magnet mentioned later, it does not cause magnetic corrosion. Thus, even when manufacturing the shape of a ring, and a tabular light-gage anisotropic magnet by suppressing contraction in the case of sintering small, processing for correcting a configuration becomes unnecessary and low-cost-izing and a productivity drive are realized. Moreover, since anti-chip box reinforcement is high, handling becomes easy and the crack between a forming cycle and a sintering process and generating of a chip of a high density Plastic solid decrease.
[0013] In this invention, in order to form the above-mentioned close hole, two alloying methods are used. Two alloying methods in R-T-B system sintered magnet manufacture are the approaches of mixing and sintering the powder of two sorts of alloys with which presentations differ. In this invention, the above-mentioned hardener for the main phases and the above-mentioned hardener for grain boundary phases are used in two alloying methods. Particle diameter is large although the thing and presentation which use the powder of the hardener for the main phases used by this invention with two conventional alloying methods are the same. And in this invention, R rich powder of the major diameter which is not in the former so that a close hole may be formed at the time of baking is used as hardener powder for grain boundary phases. This hardener powder for grain boundary phases has a low-melt point point presentation centering on Nd89Fe11 (weight ratio). The powder of the hardener for grain boundary phases is fused at the time of sintering, to the R2 T14B main phase, serves as the wettability very good liquid phase, flows, covers the perimeter of the powder of the hardener for the main phases, serves as a magnetic grain boundary phase, and raises coercive force. The powder of the hardener for grain boundary phases is a major diameter, and, moreover, it is easy to do melting and floating of it. For this reason, after the hardener powder for grain boundary phases carries out melting floating, the big close hole which is not buried with a sintering reaction is left behind.
[0014] Although R rich powder which serves as a grain boundary phase after sintering is added also with two conventional alloying methods, since the conventional R rich powder is a minor diameter, a close hole does not remain in a sintered compact. First of all, R rich powder is added for promoting liquid phase sintering and achieving magnetic densification with two conventional alloying methods,

while raising coercive force. In two alloying methods which add R rich powder, the proposal of lowering a sintered compact consistency and reducing contraction is not made conventionally.
[0015] Although an open hole also exists near the front face of the sintered magnet of this invention, if a part of sintering process [at least] is performed in a vacuum or under a reduced pressure ambient atmosphere, in order that the liquid-phase-ized hardener for grain boundary phases may take up the free passage way to the exterior of an open hole, the rate of an open hole decreases and corrosion resistance improves.
[0016] What (the consistencies after sintering are 7.15 g/cm3 following) is not made to sinter thoroughly in this invention, using the Plastic solid of high density (5.5 g/cm3 above) is desirable. Thereby, contraction at the time of sintering becomes still smaller.
[0017] Although the magnetic properties {(BH) max =about 17 to 25 MGOe} of the sintered magnet manufactured by this invention become lower than the conventional R-T-B system high density sintered magnet, they become higher than the BONDIDDO magnet {(BH) max =about 15 MGOe (s)} of a Sm-Co system. A R-T-B system magnet has a cheap raw material compared with a Sm-Co system magnet. Therefore, the sintered magnet manufactured by this invention is suitable conventionally as a substitute of the Sm-Co system BONDIDDO magnet used for the light-gage magnet.
[0018] In addition, although it is known as are shown below, and how to manufacture a sintered compact with a porous low consistency, without making various kinds of proposals which manufacture an R2 T14B system sintered magnet with two alloying methods, and sintering a Plastic solid thoroughly is shown below, each of these differs from this invention.
[0019] The manufacture approach of an anisotropy rare earth bond magnet is indicated by JP,5-47528,A. After mixing a sintering inhibition agent or an evaporation agent to Nd-Fe-B magnet powder or oxidizing the front face of magnet powder first by this approach, magnet powder is set in a field, and it is 0.2 - 5 t/cm2. It compresses by the pressure and a green compact is made. Subsequently, the anisotropy baking object which calcinates a green compact at 500-1140 degrees C, and has an open pore is made, and it heat-treats at 400-1000 degrees C. Subsequently, resin is hardened after sinking resin into an open pore. Various sintering inhibition agents are added in the tables 1-2 of this official report, the consistency of the baking object (before resin impregnation) calcinated and manufactured is indicated at 700-1060 degrees C, and each of these is 6.9 g/cm3. It has become the following.
[0020] A sintering inhibition agent given [this] in an official report stops at extent which does not fuse an oxide, a fluoride, a chloride, etc. during baking, or is fused in part. R which these sintering inhibition agents produce in this official report at the time of baking -- since floating of the rich liquid phase is barred, even if it performs elevated-temperature baking, a baking object stops contracting greatly consequently, burning temperature can be made higher than before, and it is supposed that high coercive force will come to be acquired. Evaporation agents given [this] in an official report are camphor, Lynn, sulfur, tin, etc., and these are evaporated during baking and make an open pore remain. An open pore is the continuation pore of the magnitude into which the inlet port of a hole is shown in the front face of a baking object, and resin can invade.
[0021] With the approach of this official report, they are 6.9 g/cm3. Although the following low consistency sintered magnets are obtained, it is the object that the approach of this official report forms an open pore unlike this invention. Before the closed pore is formed in this official report, it has the publication of the purport which stops baking, and the publication of such a good purport that the volume of the open pore to the total hole volume is comparatively (effective porosity) high, and the technical thought of this invention of making the ratio of a close hole high is not seen. Since a sintered magnet given [this] in an official report makes an open pore a subject, resin impregnation is indispensable because of corrosion-resistant reservation, and since it is necessary to make resin reach into the open pore moreover prolonged to the magnetic depth section, productivity will become remarkably low. For example, in the example of this official report, after performing resin impregnation for 2 hours after carrying out vacuum suction, pressurizing further and performing impregnation of 2 hours, hardening processing of resin has taken 2 hours.
[0022] In order to form a close hole in this invention, since R rich powder of a predetermined presentation is added, coercive force also improves, but by the approach of this official report, since

the open pore is formed using the above sintering inhibition agents or an evaporation agent, distribution of R in the inside of a magnet becomes poor, and coercive force serves as imperfection. On the other hand, if the magnetic amount of R is made to increase for the improvement in coercive force, a residual magnetic flux density will become imperfection. There is no publication about the dimension of a sintering inhibition agent in this official report. In addition, this official report has the publication of the purport which may add Tb and the metal powder of Dy in the range in which contraction of a baking object does not become not much large for the improvement in coercive force. However, the effectiveness as the hardener powder for grain boundary phases with the low melting point which the melting point of 1357 degrees C and Metal Dy is 1407 degrees C, and uses it by this invention that the melting point of Metal Tb is the same is not acquired. And the particle diameter range of the metal Tb metallurgy group Dy is not indicated by this official report, but there is also no example which added these.

[0023] moreover, the mean particle diameter with a Nd-Fe-B alloy desirable in this official report -- 2-20 micrometers it is -- ** -- it indicates -- having -- **** -- an example -- 3.5 micrometers Impalpable powder is used. The pressure applied in the case of powder compacting although the consistency of the green compact before baking is not indicated by this official report is 0.2 - 5 t/cm2. It is low voltage and it is thought that the high density Plastic solid is not acquired. Also at these points, an approach given [this] in an official report differs from this invention.

[0024] Although the approach of sintering the mixture (mean particle diameter of 3-7 micrometers) of the end of a Nd-Fe-B alloy powder and the end of a Nd-Co alloy powder is indicated by JP,60-230959,A, at the example of this official report, it is consistency 7.4 g/cm3. The precise sintered magnet is produced and it completely differs from this invention which forms a close hole.

[0025] the approach of sintering the mixture of the end of a R-T-B system alloy powder and the end of a R-X (X -- Fe or Fe, and one or more sorts of B, aluminum, Ti, V, Co, Zr, Nb, and Mo of mixture) alloy powder is indicated by JP,63-93841,A. It is manufactured by quenching melt and is ****** as sintering acid this end of a R-X alloy powder. At the example of this official report, it is 1 t/cm2. It fabricates, sinters at 1000-1200 degrees C, and is consistency 7.43 g/cm3. The precise sintered magnet is manufactured. In this official report, it sets at an example, and is 1-500 micrometers. The sintered magnet obtained in the example although there was a publication of the purport using the end of a R-X alloy powder is consistency 7.43 g/cm3. It is precise. The technical thought of suppressing the contraction at the time of sintering by daring form a hole in this official report is not seen.

[0026] In the approach of manufacturing an R2 T14B system magnet alloy with powder-metallurgy processing, sintering the powder-molding object which does 0-70 volume % content of the end of an alloy powder it is obtained from the end of a liquid quenching alloy powder or thin band (amorphous and microcrystal) which has the presentation Pr, Tb, and whose Dy value are 32 - 100 % of the weight is indicated by JP,63-278208,A. R rich powder used in the example of this official report although this approach is two alloying methods which use R rich powder is the mean particle diameter of 3-5 micrometers. Since it is impalpable powder, a close hole is not formed at the time of sintering.

[0027] The approach of mixing and sintering A alloy which consists of R2 T14 B phase, and B alloy which contains R, CoFe, and B and has R rich phase is indicated by JP,5-21219,A. the example of this official report -- both alloys -- mean particle diameter of about 5 micrometers up to -- the sintered compact which is pulverized and was obtained -- all -- consistency 7.42 g/cm3 More than is precise and it completely differs from this invention.

[0028] The manufacture approach of a compound-die magnet ingredient of having the mixed process which mixes the matrix material powder containing a low-melt point point element (at least one sort of aluminum, Zn, Sn, Cu, Pb, S, In, Ga, germanium, and Te) or a high-melting element and R2 T14B system magnetism powder in JP,63-114939,A, and forms mixed powder in it, and a magnet chemically-modified [which fabricates and magnet-izes said mixed powder] degree is indicated. And the process which fabricates and sinters mixed powder as a magnet chemically-modified [said] degree, or the heat application-of-pressure process which gives application of pressure between heat to mixed powder, and generates a Plastic solid is mentioned. In addition, before the application of pressure between heat, preforming is performed preferably. temperature with it -- it is -- heat

application-of-pressure temperature -- 300-1100 degrees C and a heat application-of-pressure pressure -- 5 - 5000 kgf/cm2 it is . [sintering temperature higher than the melting point of a matrix material, and] [lower than 1150 degrees C] In this official report, it is making to raise the dimension yield into the technical problem, and this official report has description of the purport which can raise the dimension yield of a product by the hot-forming method. However, at the example of this official report, all the consistencies after sintering or the application of pressure between heat are 7.1 g/cm3. It is the above and there is no disclosure of the consistency of the Plastic solid before sintering or the application of pressure between heat. The mean particle diameter of the R2 T14B system magnetism powder in the example of this official report is 3-4 micrometers. The particle size of the matrix material powder which is a minor diameter and contains a low-melt point point element is 20-30 micrometers at the maximum. It is a minor diameter. In this official report, it is the mean particle diameter of 100 micrometers. Although there is an example of a comparison which used aluminum for the matrix material and performed pressing between heat, it is consistency 7.5 g/cm3 in this case. The precise magnet is obtained. Each of pressures at the time of shaping in the example of this official report and pressures at the time of preforming is 1.5 t/cm2. It is as small as the following.

[0029] After carrying out press forming of the magnet powder in the approach of manufacturing a RFeB system magnet with powder-metallurgy processing, it considers as a porous sintered compact in a 400-900-degree C temperature requirement, and the approach of carrying out fixed time amount immersion of it at the melting alloy Ndx Fe 1-x (x=0.65-0.85) is indicated by JP,3-80508,A. This approach aims at suppressing the deformation after sintering resulting from the anisotropy of the heat shrink by magnetic field orientation. However, this approach is not two alloying methods. Moreover, the Nd2 Fe14B magnet powder used in the example of this official report is about 10 micrometers. It is a minor diameter and compacting pressure, the consistency of a Plastic solid, and the consistency of a sintered compact porous [after low temperature sintering] are not indicated by this official report.

[0030] In case the 2-17 system magnet of Sm2 Co17 or Pr2 Co17 grade is manufactured, the approach of sinking in a Plastic solid after a temporary-quenching join at 400-900 degrees C, and sinking in liquefied plastics is indicated by JP,55-15224,A. This approach aims at the improvement in on the strength of a magnet. In the example of this official report, it is 5-30 micrometers. It is indicated that contraction when carrying out full sintering at that contraction when fabricating a particle and sintering at 800 degrees C was 7% and 1150 degrees C was about 12 - 15%. and the consistency after being immersed in an epoxy resin and solidifying a temporary sintered compact -- 6.80 g/cm3 it was -- things are indicated. However, this approach differs not in two alloying methods but in a magnet presentation from this invention. With this official report, it is 5-30 micrometers. The minor diameter particle is used and the consistency of the Plastic solid in front of a temporary-quenching join is not indicated by this official report.

[0031] It is 200 micrometers about the ingot which carried out solution treatment of the Nd-Fe-B system alloy ingot to JP,62-281307,A in the 1000-1150-degree C temperature requirement, and carried out solution treatment to it. It grinds to the following particle size and the approach of annealing the pulverized Plastic solid in the end of an alloy powder in a 500-1050-degree C temperature requirement and the approach of making carry out impregnation of the plastics to the Plastic solid which annealed, and solidifying are indicated. In this approach, it anneals for removing a crushing strain and raising coercive force at 500-1050 degrees C. In the example of this official report, it is annealing, after fabricating the alloy-powder end of a minor diameter (mean particle diameter of 5 micrometers) by the low voltage force (2 t/cm2). The consistency of a Plastic solid and the consistency of a sintered compact are not indicated by this official report.

[0032] After grinding the alloy which makes a fundamental component rare earth elements, iron, and boron and fabricating it among a magnetic field, it sinters in JP,4-314307,A and the method of manufacturing the bulk object for bond magnets is indicated. By this approach, the bulk object of partial-loss-by-fire joint gold with 60 - 95% of consistency of theoretical density is manufactured by sintering at the temperature of 700-1000 degrees C for 3 or less hours. Partial-loss-by-fire joint gold is an organization which includes a hole considerably, and since a hole serves as a nucleus of crack development, and a nucleus of further destruction, it can be easily ground with small stress.

Therefore, the mechanical distorted effect of [at the time of crushing] decreases. At the example of this official report, it is 3 micrometers of mean diameters. After fabricating pulverized coal, a partial-loss-by-fire join is carried out, and the bulk object is manufactured. The consistency of a Plastic solid and contraction at the time of a partial-loss-by-fire join are not indicated by this example. Invention given [this] in an official report differs from this invention not using two alloying methods in that pulverize the bulk object of partial-loss-by-fire joint gold, and a bond magnet is manufactured. The consistencies of the bulk object of the partial-loss-by-fire joint gold in the example of this official report are 5.6 g/cm3. It is the following and comparable as the Plastic solid consistency in this invention. Therefore, since past [the high one], magnetic properties, and reinforcement run short of void contents, the bulk object of partial-loss-by-fire joint gold given [this] in an official report cannot be used as a bulk magnet. That is, grinding and the formation of a bond magnet are indispensable. For this reason, coercive force will deteriorate and a manufacturing cost will become high.

[0033] Moreover, after fabricating the bulk object of partial-loss-by-fire joint gold given in JP,4-314307,A among a magnetic field, the method of carrying out impregnation of the resin to a Plastic solid, and manufacturing a bond magnet is indicated by JP,4-314315,A. Shaping in this approach among a magnetic field serves both as grinding and shaping of a bulk object of partial-loss-by-fire joint gold. The anti-chip box reinforcement of the sintered compact of the former [official report / this] is 2.5 t/cm2. The anti-chip box reinforcement of the bulk object of partial-loss-by-fire joint gold is 1 t/cm2 to being above. It is dramatically as small as the following and the purport that grinding is easy is indicated. At the example of this official report, it is 3 micrometers of mean diameters like JP,4-314307,A. The partial-loss-by-fire join of the pulverized coal is fabricated and carried out, and it is consistency 5.2 g/cm3. The following bulk objects are manufactured, and it presses further, and carries out resin sinking in, and they are a consistency 5.9 - 6.0 g/cm3. The bond magnet is manufactured. Since the consistency is still lower, the bulk object of partial-loss-by-fire joint gold given [this] in an official report is more nearly impossible for using it as a bulk magnet, without performing compression molding and resin impregnation than partial-loss-by-fire joint gold given in JP,4-314307,A. For this reason, coercive force will deteriorate and a manufacturing cost will become high.

[0034] At conventional partial-loss-by-fire joint gold which was described above, it is 30 micrometers with the 2-17 system magnet of Sm2 Co17 grade. Although there is an example for which the powder which consists of a particle is used, with an R2 T14B system magnet, it is the mean particle diameter of 3 micrometers. The partial-loss-by-fire join of the Plastic solid of the magnet powder which consists of a minor diameter particle of order is carried out. Although it is necessary to heat-treat at temperature lower than the time of performing full sintering when carrying out the partial-loss-by-fire join of the Plastic solid which consists of such a minor diameter particle, in a low temperature field, a sintered compact consistency will change a lot corresponding to change of retention temperature. That is, in order to manufacture the half-sintered compact of a predetermined consistency, strict temperature management will be needed and a manufacturing cost will rise.

[0035] On the other hand, in this invention, it differs from the approach conventional at the point of using first two alloying methods which use R rich powder of a major diameter. Moreover, it differs in that the thing of a major diameter is used for the powder used as the magnetic main phase. In the Plastic solid containing the powder for the main phases of a major diameter, since the particle migration through rare-earth-elements Rich's liquid phase is difficult, even if the retention temperature in a sintering process is an elevated temperature (for example, the conventional full sintering temperature field), a sintering reaction will not advance, before carrying out full sintering. For this reason, the sintered compact of a predetermined low consistency will be stabilized, and will be obtained in a large temperature requirement, and management of a sintering process becomes very easy. Moreover, since it is hard to condense the particle of a major diameter, handling becomes easy, especially it becomes easy at the time of shaping to fill it up to metal mold.

[0036]

[Elements of the Invention] Hereafter, the concrete configuration of this invention is explained to a detail.

[0037] The sintered magnet of <sintered magnet> this invention contains R (R is at least one sort of the rare earth elements containing Y), T (T is Fe, or Fe and Co), and B.
[0038] Although especially a magnet presentation is not limited, B is contained for R 0.5 to 3.5% of the weight 30 to 45% of the weight, and it is usually desirable that the remainder is T substantially.
[0039] It is Y, a lanthanide, and actinide, as R, among Nd, Pr, and Tb, at least one sort, especially Nd of R are desirable, and it is desirable that Dy is included further. Moreover, one or more sorts in La, Ce, Gd, Er, Ho, Eu, Pm, Tm, Yb, and Y may also be included. Mixture, such as a misch metal, can also be used as a raw material of rare earth elements. The phase which is rich in iron when there are too few R contents deposits, high coercive force is no longer acquired, and if there are too many R contents, a high residual magnetic flux density will no longer be obtained.
[0040] If there are too few B contents, high coercive force will no longer be acquired, and if there are too many B contents, a high residual magnetic flux density will no longer be obtained.
[0041] In addition, as for the amount of Co(es) in T, it is desirable to consider as 30 or less % of the weight.
[0042] In order to improve coercive force, elements, such as aluminum, Cr, Mn, Mg, Si, Cu, C, Nb, Sn, W, V, Zr, Ti, and Mo, may be added, but if an addition exceeds 6 % of the weight, lowering of a residual magnetic flux density will pose a problem.
[0043] In the magnet, carbon and oxygen may contain as others, an unescapable impurity, or a minute amount additive. [elements / these]
[0044] The sintered magnet of this invention has the main phase of the crystal structure of tetragonal system substantially, and R rich phase with high R ratio exists in a grain boundary rather than R2 T14B. The magnetic diameter of average crystal grain becomes a thing according to the diameter of crystal grain and sintering conditions of the hardener for the main phases which are mentioned later.
[0045] The sintered magnet of this invention includes a close hole. A close hole is a hole which is not open for free passage on a magnet front face. A close hole is three to 15 magnetic volume %, and is three to 12 volume % preferably. The magnet which has too few close holes is greatly contracted at the time of sintering, and dimensional accuracy with a good Plastic solid is not maintained. The magnet which has too many close holes becomes inadequate [a magnet property], and also runs short of reinforcement. The sum total floor area ratio of the close hole in a magnet and the sum total floor area ratio of an open hole mentioned later are computable as follows.
[0046] Open hole sum total floor area ratio K type I K=(WW-W)/V close hole sum total floor area ratio H type II H=1-K-W/(V-rho)
However, volume, W for which it asked from V:sample configuration in each above-mentioned formula: Sample weight, WW : Sample weight after being immersed underwater, decompressing a sample to 100 or less Torrs, holding it for 30 seconds and wiping off the water on ejection and the front face of a sample, rho: It is magnetic theoretical density.
[0047] although especially the configuration and dimension of a close hole are not limited -- the average projection cross section per close hole -- 1000-30000 micrometers 2 it is -- things are desirable. Even when a small close hole is formed in early stages of sintering, in order to disappear by sintering termination, generally the average projection cross section of a close hole is 1000 micrometers. 2 It is hard to become the following. That is, the average projection cross section is 1000 micrometers. 2 If it is going to form the close hole of the following, sintering will progress too much, without forming a close hole, the sum total volume of a close hole will become small, and contraction will not become small. Moreover, since the crystal grain with which the magnetic consistency of the crystal grain contiguous to a close hole is the same although coercive force becomes small, and it adjoins a close hole when the average volume per close hole is small increases in number, high coercive force is hard to be acquired. On the other hand, if the average projection cross section is too large, magnetic reinforcement will serve as imperfection. Moreover, the average projection cross section is 30000 micrometers. 2 Since it is necessary to spend the hardener for grain boundary phases huge in order to form the close hole which exceeds, with a light-gage magnet, shaping becomes difficult and magnetic surface magnetic flux tends to become uneven. The cross section of a close hole can be measured using the scanning electron microscope photograph of a magnet cross section. A photograph is taken after grinding a cutting plane after cutting a magnet on the occasion of measurement, and forming the golden spatter film in a cutting plane further. And the

cross section is measured about 100 or more close holes of arbitration per magnet, and the average is calculated, and let this be the average projection cross section per close hole.

[0048] The consistency of the sintered magnet of this invention is 7.15 g/cm3. It is desirable that it is the following. 200 micrometers If it fabricates with high voltage using the particle of the major diameter of extent, it will be the consistency of a Plastic solid 6.4 g/cm3 Even if it calcinates at an elevated temperature with such a Plastic solid in the case of baking since particle migration is difficult although it can be made high with extent, they are 7.15 g/cm3. It is difficult to consider as the consistency which exceeds. On the contrary, when it considers as the Plastic solid of a low consistency using the particle of a minor diameter, they are 7.15 g/cm3. If it calcinates until it becomes the consistency which exceeds, sintering will progress too much and contraction will become large. Even if the consistency of a sintered magnet is this range, when there are many open holes which are open for free passage on a magnet front face, since magnetic corrosion resistance falls extremely, it is not desirable. As for the ratio of an open hole, it is desirable that it is below 2 volume %. It can ask for the ratio of an open hole by the approach mentioned above.

[0049] As for the sintered magnet of this invention, manufacturing by the approach shown below is desirable. By this approach, in a forming cycle, the Plastic solid of the mixture of the powder of the hardener for the main phases and the powder of the hardener for grain boundary phases is manufactured, and said Plastic solid is sintered in a sintering process.

[0050] Although what is necessary is just to determine suitably the presentation of the hardener for the <hardener for main phases> main phases in consideration of a presentation and its mixed ratio of the hardener for grain boundary phases according to the magnet presentation made into the object, B is contained for R 0.5 to 3.5% of the weight 26 to 35% of the weight, and it is usually desirable that the remainder is T substantially.

[0051] although a sintering reaction advances by R rich phase's turning into the liquid phase, and flowing with an R2 T14B system magnet -- this invention -- R -- since it is necessary to suppress progress of a sintering reaction in order to add the rich hardener powder for grain boundary phases and to suppress contraction, as for R content of the hardener for the main phases, lessening is desirable.

[0052] The hardener for the main phases has the main phase mentioned above and R rich phase mentioned above. Especially the diameter of average crystal grain of the powder of the hardener for the main phases is not limited. Since it is desirable that it is the diameter of crystal grain which serves as a single crystal particle when it considers as the particle diameter mentioned later since it anisotropy-izes by magnetic field orientation, but crystal grain should just be carrying out orientation within the particle in this invention even if it is a polycrystal particle, the diameter of average crystal grain is 3-600 micrometers. It can choose from the range where extent is large.

[0053] The mean particle diameter of the powder of the hardener for the main phases is 20 micrometers preferably. It is 50-350 micrometers more preferably above. It carries out. If mean particle diameter is too small, the effectiveness by the formation of a particle major diameter mentioned above will serve as imperfection. On the other hand, if mean particle diameter is too large, in the Plastic solid of thin meat, magnetic field orientation will become difficult. In addition, the mean particle diameter of the hardener powder for the main phases computes the average projected area per particle, and makes it the diameter when converting this into a circle. Especially the measuring method of the projected area of a particle is not limited. For example, powdered dispersion liquid can be applied on a glass plate so that particles may not lap, a photograph can be taken, and it can ask for the projected area of a particle from this photograph. In addition, it can also ask for the projected area of a particle by scanning said spreading object by the light beam, and detecting reflection factor change.

[0054] Especially the manufacture approach of the powder of the hardener for the main phases may not be limited, but may use any, such as the approach of carrying out disintegration of the casting alloy by hydrogen absorption grinding etc., and a reduction diffusion method, and may grind and carry out disintegration of the sintered magnet. If the sintered magnet anisotropy-ized by magnetic field orientation is ground, since the polycrystal particle of a major diameter which consists of crystal grain of the minor diameter by which orientation was carried out can be obtained, the magnet of a high residual magnetic flux density and high coercive force is obtained.

[0055] The remainders of the hardener for <hardener for grain boundary phases> grain boundary phases are Fe and/or Co substantially 75 to 92% of the weight preferably, including R 70 to 97% of the weight. As R contained in the hardener for grain boundary phases, Nd is desirable, it is more desirable that Nd occupies 50% or more in R, and it is still more desirable to use only Nd substantially as R. If there are few amounts of Nd(s) in R and there are few amounts of R, the melting point of the hardener for grain boundary phases will not become low, but a close hole will become is hard to be formed. Although 640 degrees C and Nd81Co19 (weight ratio) eutectic alloy of the melting point of Nd89Fe11 (weight ratio) eutectic alloy are 566 degrees C, the melting point of Dy88Fe12 (weight ratio) eutectic alloy is 890 degrees C. The hardener for grain boundary phases used by this invention does not contain B. B in the hardener for grain boundary phases does not contribute to improvement in a magnet property, and does not contribute to lowering of the melting point of the hardener for grain boundary phases, either.
[0056] For the powder of the hardener for grain boundary phases used by this invention, an aperture is 38 micrometers. An aperture is 53 micrometers preferably above. It remains to the above sieve and an aperture is 500 micrometers. An aperture is 250 micrometers preferably hereafter. The following sieves are passed. If the particle diameter of the powder of the hardener for grain boundary phases is small, the magnet which has a predetermined close hole will no longer be obtained, and also the powder of the hardener for grain boundary phases becomes being easy to oxidize. If the particle diameter of the hardener for grain boundary phases becomes large too much, a hole will become large too much and surface magnetic flux will tend to become uneven. Moreover, if the dimension of the hole which remains in a magnet becomes large too much to a magnet dimension, sufficient magnet reinforcement will no longer be obtained.
[0057] Although especially the manufacture approach of the hardener for grain boundary phases is not limited, a melt quenching method is used preferably. The approach of contacting an alloy molten metal to a cooling base, and cooling as a melt quenching method, for example, the single rolling method, the congruence rolling method, and revolution disc method ****** are desirable, and may use the gas atomizing method. Cooling of an alloy molten metal is performed in non-oxidizing atmospheres, such as nitrogen and Ar, or a vacuum. When a cooling rate is slow, phase separation of the hardener for grain boundary phases of the above-mentioned presentation will be carried out mainly to Nd and Fe2 Nd. These melting points are as high as 1000 degrees C or more, and in order that Nd may tend [very] to oxidize, close hole formation becomes difficult. The hardener for grain boundary phases manufactured by the melt quenching method has an amorphous phase or a microcrystal phase.
[0058] Especially the manufacture approach of the mixture of the powder of the hardener for the <grinding process and mixed process> main phases and the powder of the hardener for grain boundary phases is not limited. For example, after grinding simultaneously, manufacturing mixture, after mixing both hardeners, and grinding each hardener, both hardeners may be mixed and mixture may be manufactured by pulverizing further if needed.
[0059] The ratio of the hardener for grain boundary phases in mixture is more preferably made into 3 - 12 % of the weight two to 20% of the weight. If this ratio is too low, it will become difficult to form close hole sufficient in a magnet, and if this ratio is too high, it will become difficult to obtain the magnet of a high property.
[0060] Especially the grinding approach of each hardener is not limited, but may grind combining these that what is necessary is just to choose suitably the mechanical grinding method, the hydrogen absorption grinding method, etc. However, since the sharp magnet powder of particle size distribution is obtained, it is desirable to perform hydrogen absorption grinding. It is desirable to use air-current type grinders, such as a jet mill, for it, since sharp particle size distribution are acquired by mechanical grinding.
[0061] The mixture of the powder of both hardeners is fabricated in a <forming cycle> forming cycle all over a magnetic field. At this time, the consistency of a Plastic solid is desirable and they are 5.5 g/cm3. They are 6.0 g/cm3 more preferably above. It fabricates so that it may become the above. In a Plastic solid with a small consistency, if it is going to acquire sufficient magnet property, contraction at the time of sintering will become large, and if contraction at the time of sintering is made small, a magnet property will become imperfection. Although there is especially no upper limit

of the consistency of a Plastic solid, it is difficult to consider as the consistency exceeding 6.4 g/cm3. For example, they are 20 t/cm2 at the time of shaping. Since the above extra-high voltage is needed, a shaping equipment metallurgy mold will become expensive, and the configuration of a Plastic solid will be restricted to a simple thing. Although utilization of a lot of organic lubricant is also effective in order to raise a Plastic solid consistency, it is difficult before sintering to remove organic lubricant, and the carbon residue in a magnet will reduce a magnet property. In addition, the consistency of a Plastic solid is computable from the dimension of the Plastic solid measured by a micrometer etc.

[0062] Thus, for the Plastic solid of a high consistency, anti-chip box reinforcement is 2 0.3 kgf (s)/mm. In a pan, they are 0.5 kgf/cm2 above. Since it becomes the above, handling becomes easy and generating of a crack or a chip decreases.

[0063] Especially compacting pressure is 8 t/cm2 preferably, although what is necessary is just to determine suitably that it is not limited but the Plastic solid of a desired consistency is acquired. It is 2 12t/cm more preferably above. It considers as the above. Magnetic field intensity at the time of shaping is usually preferably carried out to more than 15 kOe more than 10 kOe.

[0064] The field impressed at the time of shaping may be a direct-current field, or may be a pulsed magnetic field, and may use these together. The pressure impression direction and the field impression direction can apply this invention also to the so-called, vertical magnetic field fabricating method mostly in agreement also at the so-called horizontal magnetic field fabricating method the pressure impression direction and the field impression direction intersect perpendicularly mostly.

[0065] <Sintering process> The Plastic solid acquired as mentioned above is sintered and magnetized.

[0066] The values (consistency variation at the time of sintering) which subtracted the consistency of a Plastic solid from the consistency of a sintered compact in this invention are 0.2 g/cm3. Sintering so that it may become the above is desirable. When consistency change at a sintering process is too small, sintering is inadequate and a magnet property and a mechanical strength serve as imperfection. In order to make contraction small, it is consistency variation more preferably three or less 1.5 g/cm 1.2 g/cm3 It considers as the following.

[0067] What is necessary is just to choose suitably so that there may be especially no limit in the various conditions at the time of sintering and the consistency change at the time of sintering etc. may serve as a desired value. Although the retention temperature at the time of sintering should just be more than the melting temperature of the hardener for grain boundary phases, as mentioned above, since it forms a low consistency magnet by using the hardener powder for grain boundary phases of a major diameter, it can make retention temperature higher than the case of the so-called conventional partial-loss-by-fire join by this invention. At 900-1100 degrees C, heat treatment is performed for 0.5 to 10 hours, it sinters, and, specifically, quenching is desirable after that. In addition, it is desirable that they are inert gas ambient atmospheres, such as inside of a vacuum or Ar gas, and a sintered atmosphere is the point that the ratio of an open hole can be reduced, as mentioned above, and sintering in the inside of a vacuum or the decompressed inert gas ambient atmosphere is more desirable [a sintered atmosphere]. In addition, it is good also as a configuration which makes a part of sintering process a vacuum or a reduced pressure ambient atmosphere.

[0068] Aging treatment is performed after <other> sintering if needed for the improvement in coercive force.

[0069] In order to raise magnetic corrosion resistance, it is desirable to take up an open hole. What is necessary is just to perform processing to dry, after for that a magnet is immersed into the solution which dissolved resin in the organic solvent. In addition, the usual corrosion-protective covering may be prepared with electropainting, nonelectrolytic plating, etc. of resin after such processing.

[0070] This invention is suitable for manufacture of the shape of a ring of thin meat, or a tabular magnet which is mentioned later, and this invention is suitable for manufacture of the light-gage magnet especially whose thickness is 3mm or less. In addition, when magnet thickness is set to less than 0.5mm, there is an inclination for shaping to become difficult.

[0071] In <dimension deflection> this invention, since the very small sintered magnet of dimension deflection is obtained, it can produce commercially after sintering, without carrying out configuration processing by grinding etc.

[0072] That is, according to this invention, it has a parallel part and thickness deflection of a parallel part can be made into 1.5% or less in the light-gage sintered magnet whose value which **(ed) length between couplings of a parallel part by the average thickness is ten or more, considering as 1% or less is also easy, and it is possible to store thickness deflection in such range also about the light-gage magnet whose length between couplings / average thickness are 15 or more. A parallel part is the block inserted by the 2nd parallel page which counters, and the magnet which has a parallel part is a tabular magnet [for example,], disc-like magnet, and ring-like magnet. The thickness deflection of a parallel part is the value which **(ed) the difference of the maximum of the thickness of a parallel part, and the minimum value with the length between couplings of a parallel part. The thickness deflection of a parallel part is a value used as the index of the curvature of a parallel part, or the heterogeneity of thickness, and since the unevenness of curvature or thickness becomes large in the case of the above light-gage sintered magnets of a proportion, generally thickness deflection is 2.5% or more conventionally.
[0073] Moreover, according to this invention, it has a body and the outer-diameter deflection and/or bore deflection of a body can be made into 1.5% or less in the light-gage magnet whose value which **(ed) the average outer diameter of a body by the average wall thickness is ten or more, considering as 1% or less is also easy, and it is possible to store outer-diameter deflection and/or bore deflection in such range also about the light-gage magnet whose average outer diameter / average wall thickness are 15 or more. Although a body is a cylindrical block which has a peripheral face or has a peripheral face and inner skin and the magnets which have a body are for example, a ring-like magnet and a disc-like magnet, the outer-diameter deflection and bore deflection in this case are aimed at the body which has a peripheral face and inner skin. The outer-diameter deflection of a body is the value which **(ed) the difference of the maximum of the outer diameter of a body, and the minimum value with the average outer diameter, and bore deflection is the value which **(ed) the difference of the maximum of the bore of a body, and the minimum value with the average bore. The outer-diameter deflection and bore deflection of a body are a value used as the curvature of a body, or the index of distorted and thick heterogeneity, and since curvature and distorted and thick unevenness become large in the case of the above light-gage sintered magnets of a proportion, generally outer-diameter deflection and bore deflection are 3% or more conventionally.
[0074] In addition, it is also easy for a disc-like magnet etc. to be able to have the body which has only a peripheral face, to be able to make outer-diameter deflection of a body into 1.5% or less also in the light-gage sintered magnet whose average outer diameter / average thickness are 10 or more and further 15 or more, and to consider as 1% or less.
[0075] In this description, the thickness deflection of a parallel part is measured as follows. First, while constitutes the parallel part, and a device under test is laid on a surface plate so that a field may touch a surface plate. And the height from the surface plate front face of the field of another side which constitutes a parallel part is measured by 20 places. Next, a device under test is turned over, it lays on a surface plate, and height is similarly measured by 20 places so that the field of said another side may touch a surface plate front face. a measuring point -- the field of the measuring object -- almost -- equal -- 20 -- dividing -- the inside of each field -- it considers as a central point mostly. From all the obtained measured value, the difference (Tmax-Tmin) of maximum (Tmax) and the minimum value (Tmin) is calculated. Let the value {(Tmax-Tmin) /L} which **(ed) this difference at the maximum L of the die length (longitudinal direction die length) of each field which constitutes said parallel part be thickness deflection. The thickness deflection of the light-gage magnet which has 2 or more sets of parallel fields mutually serves as a big value, when both principal planes are made into one [said] field and the field of said another side. In addition, what is necessary is just to use for the average thickness in explanation of a light-gage magnet the average of all the measured value obtained as mentioned above.
[0076] It asks for the outer-diameter deflection and bore deflection of a body as follows. First, the shaft orientations of a body are followed, the outer diameter or bore of a body is measured, and maximum and the minimum value are calculated. At this time, the measured value of the range of 0.1mm of the shaft-orientations both ends of a body is excepted. Next, same measurement is performed after rotating 15 degrees of said bodies centering on the shaft. Thus, measurement is repeated a total of 12 times over 180 degrees of hoop directions at intervals of 15 degrees. It is

phimin about the minimum thing among phimax and the minimum value of 12 in the greatest thing among the maximums of 12. It carries out and is phimax-phimin. It asks. Next, the average phi 0 of the average of the maximum of 12, and an average of the minimum value of 12 It asks and is phi 0. It considers as an average outer diameter or an average bore. And let {(phimax-phimin) /phi0} be outer-diameter deflection or bore deflection. In addition, in the average outer diameter in explanation of the proportion of a light-gage magnet, and an average bore, it is the above phi 0. What is necessary is just to use a (average outer-diameter-average bore) / 2 for average wall thickness that what is necessary is just to use.
[0077] In addition, a non-contact-type measuring instrument, such as optical, may be used for measurement of dimension deflection, and the measuring instrument of contact processes, such as a contact process three-dimension measuring instrument, and a micrometer, an inner circumference micrometer, may be used for it.
[0078]
[Example] Hereafter, the concrete example of this invention is shown and this invention is further explained to a detail.
[0079] The sintered magnet sample shown in the <example 1> table 1 was produced by the approach shown below.
[0080] First, the ingot of the hardener for the main phases was manufactured by casting. The presentation of an ingot is shown in a table 1. In addition, the remainder of a presentation is Fe. the diameter of average crystal grain of these alloy ingots -- 300 micrometers it was . After carrying out coarse grinding of each alloy ingot using expansion and contraction of the volume by hydrogen absorption and the degasifying reaction, the disc mill ground and it considered as the powder of the mean particle diameter shown in a table 1. In addition, it asked for powdered mean particle diameter by the approach mentioned above from the optical microscope photograph of a powdered paint film.
[0081] Next, the alloy molten metal was cooled by the single rolling method in Ar ambient atmosphere, and the hardener for grain boundary phases of the presentation shown in a table 1 was manufactured. In addition, the remainder of the presentation shown in a table 1 is Fe. Cu roll was used for the cooling roller. The hardener for grain boundary phases is thin band-like [with a thickness of 0.15mm], and it was checked as a result of the X diffraction that it is in an amorphous condition. The pin mill ground each hardener for grain boundary phases, and the end of an alloy powder it was obtained was classified by the sieve. The sieve used for the classification of each powder is shown in a table 1. In addition, the sieve with the large aperture which regulates the upper limit of particle diameter is shown in a table 1 as a passage sieve by making into a residual sieve a sieve with the small aperture which regulates the minimum of particle diameter.
[0082] Subsequently, the hardener powder for the main phases and the hardener powder for grain boundary phases were mixed. The addition (ratio of the hardener powder for grain boundary phases in mixture) of the hardener powder for grain boundary phases is shown in a table 1.
[0083] Each mixture was fabricated among the magnetic field and the disc-like Plastic solid with a diameter [of 20mm] and a thickness of 1.5mm was acquired. Magnetic field strength was set to 12 kOe, and the field was impressed so that an easy axis might serve as the thickness direction of a Plastic solid. Compacting pressure and a Plastic solid consistency are shown in a table 1.
[0084] Subsequently, it quenched, after sintering each Plastic solid in a vacuum. The time amount held to the heat treatment temperature at the time of sintering and its temperature is shown in a table 1. Aging treatment was performed at 650 degrees C after sintering and into Ar ambient atmosphere for 1 hour, and it considered as the disc-like sintered magnet sample. The consistency of each sintered magnet sample, the consistency variation at the time of sintering, a residual magnetic flux density (Br), and coercive force (Hcj) are shown in a table 1. In addition, the sample for magnetic-properties measurement which sintered and produced the Plastic solid with a diameter [of 15mm] and a thickness of 10mm was used for measurement of Br and Hcj. The manufacture conditions of the sample for magnetic-properties measurement presupposed that it is the same as that of each sample shown in a table 1 respectively except the Plastic solid dimension. Moreover, it asked by the approach which mentioned above the sum total floor area ratio of the open hole of each sample, and the sum total floor area ratio of a close hole. In addition, it is magnetic theoretical density 7.55 g/cm3 It calculated by carrying out. A result is shown in a table 1.

[0085]
[A table 1]

表 1

サンプル No.	主相用母合金 組成(重量%) R	B	平均粒子径 (μm)	粒界相用母合金 組成(重量%)	通過フルイ (μm)	残留フルイ (μm)	添加量 (重量%)	成形圧力 (t/cm²)	密度(g/cm³) 成形体	変化量	磁石	熱処理条件 温度 (℃)	時間 (hr)	閉空孔 (体積%)	開空孔 (体積%)	Br (kG)	Hcj (kOe)
1 (比較)	28.5Nd	1.10	100	—	—**	—**	—**	10	5.83	0.78	6.61	1075	2	2.1**	9.8*	9.4	3
2 (比較)	28.2Nd	1.11	55	88Nd	75	—**	10	10	5.78	1.73	7.51*	1075	5	0.8**	0.0	11.0	18
3 (比較)	28.3Nd	1.13	150	100Nd**	250	53	7	10	5.95	0.50	6.45	1050	2.5	1.0**	13.5*	8.0	3
4 (比較)	30.0Nd	1.09	6*	89Nd	425	53	5	10	4.45*	3.01	7.46*	1050	3	0.5**	0.5	11.3	17
5 (比較)	32.0Nd	1.09	125	91Nd	250	38	8	10	5.94	0.15*	6.09	875	2	1.2**	17.8*	7.1	1
6	29.0Nd	1.10	93	87Nd +8Co+5Cu	180	38	7	10	5.83	0.92	6.75	1050	3	8.5	1.7	9.2	15
7	28.5Nd	1.11	180	82Nd	250	38	10	10	6.05	0.82	6.87	1025	2	8.0	1.0	9.0	15
8	29.5Nd	1.08	30	89Nd+11Co	425	38	7	10	5.73	1.06	6.99	1050	4	7.0	0.3	9.1	11
9	29.0Nd	1.13	90	86Nd +0.5Al+3Cu	180	53	4	10	5.78	0.90	6.68	1050	4	10.2	1.5	8.6	17
10	32.0Nd	1.10	150	75Nd	250	53	14	10	6.03	1.05	7.08	1050	7	5.7	0.5	9.1	12
11	27.0Nd +1.8Dy	1.05	220	89Nd	355	53	2.5	10	6.12	0.64	6.76	975	6	9.5	0.4	9.4	12
12	32.4Nd	1.10	100	89Nd	355	63	8	5*	5.20*	1.75	6.95	1040	4	5.0	2.9*	8.8	16
13	32.4Nd	1.10	100	89Nd	355	63	8	13	6.06	0.95	7.01	1040	4	6.5	0.5	9.0	14
14	32.4Nd	1.10	100	89Nd	355	63	8	10	5.91	1.05	6.96	1040	4	6.8	0.9	8.9	15
15	28.7Nd	1.13	200	80Nd+10Dy	425	90	6	10	6.15	0.67	6.82	1075	4	9.1	0.6	9.3	21
16	30.0Nd	1.08	40	95Nd	500	106	6	10	5.85	0.65	6.50	1100	4	12.5	1.3	8.7	14

**) 本発明範囲を外れる値　*) 好ましい範囲を外れる値

[0086] Next, it asked for the thickness deflection of each sample by the approach mentioned above using the 1st class surface plate of JIS. Consequently, with this invention sample, thickness deflection is as remarkable as 0.2 - 0.8%, it was small, and there was very little curvature by the uneven contraction at the time of sintering. However, since sample No.12 had the low consistency of

a Plastic solid, sintering progressed and thickness deflection was 1.5%. If thickness deflection is small in this way in a light-gage magnet with a thickness of 1.5mm, it is possible to produce commercially without making the dimension correction by the grinding process. And as shown in a table 1, magnet property sufficient with this invention sample is acquired. In addition, on the occasion of calculation of thickness deflection, the magnetic diameter was used as length between couplings of a parallel part.

[0087] On the other hand, in comparison sample No.2, since the minimum of the particle diameter of the hardener powder for grain boundary phases was not regulated not using the residual sieve, sintering progressed too much with detailed R rich powder, and the close hole has decreased. In comparison sample No.4, since the Plastic solid of the low consistency formed using the powder of the hardener for the main phases with small particle diameter was sintered, sintering progressed too much and the close hole has decreased. Comparison sample No.2 and 4 had thickness deflection as large as 2.9 - 6.3%, and it turned out that big curvature has occurred by uneven contraction at the time of sintering. Commercial production is impossible if thickness deflection is large in this way.

[0088] Moreover, since a Plastic solid consistency was high and the consistency variation by sintering was small, thickness deflection was as small as 0.9%, but since the hardener powder for grain boundary phases was not added, the open void content has become [the close void content] low highly, and corrosion resistance is low at comparison sample No.1 which did not use two alloying methods. And coercive force is remarkably low. Since a Plastic solid consistency was high and the consistency variation by sintering was small, thickness deflection was as small as 0.8%, but since the metal Nd with the melting point high as a hardener for grain boundary phases was used, it becomes inadequate melting and flowing in the case of sintering, the open void content has become [the close void content] low highly like sample No.1, and coercive force is also remarkably low at comparison sample No.3. Since it sintered at low temperature, the consistency variation by sintering becomes remarkably small, the open void content has become [the close void content] low highly like sample No.1, and coercive force is also remarkably low at comparison sample No.5.

[0089] Next, after cutting each sample and grinding a cross section, the golden spatter film was formed in the cross section, the scanning electron microscope photograph was taken, and it asked for the average projection cross section per close hole. About sample No.7, the cross-section photograph with which dilation ratios differ is shown in (a) of drawing 1 , and (b). The close hole formed in this drawing of melting and floating of the flake-like hardener powder for grain boundary phases is accepted. The measurement size of a close hole carried out to 100 per each sample. consequently -- this invention sample -- the average projection cross section of a close hole -- 1500-25000 micrometers 2 it was -- a thing -- receiving -- comparison sample No.1, and 3 and 5 -- 100-700 micrometers 2 and No.2 -- 80 micrometers 2 and No.4 -- 5 micrometers 2 it was .

[0090] In addition, consistencies are 5.5 g/cm3. The above Plastic solid is 2 0.45 kgf/mm. The above anti-chip box reinforcement high enough was shown. On the other hand, with the Plastic solid for sample No.4 manufacture (consistency 4.45 g/cm3), anti-chip box reinforcement is 2 0.15 kgf (s)/mm. It was low.

[0091] Except having made the <example 2> configuration into the shape of a ring, the example 1 reached sample No.4, respectively like 9, it reached sintered magnet sample No.104 and 109 was produced. Plastic solid consistencies are 5.76 g/cm3 in 4.43 g/cm3 and sample No.109 at sample No.104. Although it became, and reached sample No.4, respectively and became a little small from 9, the consistency variation by sintering reached sample No.4, respectively, and was the same as 9. Each dimension of a Plastic solid was made into the outer diameter of 30mm, the bore of 27mm, the thickness of 1.5mm, and height of 7mm, and on the occasion of shaping, the field was impressed so that an easy axis might serve as the direction of a path.

[0092] About these ring-like sintered magnet samples, outer-diameter deflection and bore deflection were measured by the approach mentioned above. On the occasion of measurement, each sample was laid so that a peripheral face might touch on the 1st class surface plate of JIS, outer-diameter deflection is a contact process three-dimension measuring instrument, and bore deflection was measured by the inner circumference micrometer. Consequently, although outer-diameter deflection is 0.30%, bore deflection is 0.32% and the very small value was acquired in sample No.109 by this invention, outer-diameter deflection reached also to 5.5%, and 4.5% and bore deflection had it.

[impossible for commercial production at sample No.104 which sintered the Plastic solid with a low consistency]
[0093] The effectiveness of this invention is clear from the result of the above example.

[Translation done.]

TECHNICAL FIELD

[Industrial Application] This invention relates to the rare earth sintered magnet with small contraction and its manufacture approach at the time of sintering.

[Translation done.]

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CLAIMS

[Claim(s)]
[Claim 1] The sintered magnet which is a sintered magnet containing R (R is at least one sort of the rare earth elements containing Y), T (T is Fe, or Fe and Co), and B, and is characterized by 3-15 volume % Including a close hole.
[Claim 2] Consistencies are 7.15 g/cm3. Sintered magnet of claim 1 which is the following.
[Claim 3] the average projection cross section per close hole -- 1000-30000 micrometers 2 it is -- sintered magnet of claims 1 or 2.
[Claim 4] One sintered magnet of claims 1-3 whose ratios of an open hole are below 2 volume %.
[Claim 5] One sintered magnet of claims 1-4 whose remainders B is contained for R 0.5 to 3.5% of the weight 30 to 45% of the weight, and are T substantially.
[Claim 6] R (R is at least one sort of the rare earth elements containing Y), T (T) The sintered magnet which contains B and it is Fe, or Fe and Co After fabricating the mixture of the powder of the hardener for the main phases, and the powder of the hardener for grain boundary phases, Are the approach of manufacturing by sintering and said hardener for the main phases has the crystal grain which consists of R2 T14B substantially. Mean particle diameter is 20 micrometers. Are above and said hardener for grain boundary phases contains R 70 to 97% of the weight. The remainders are Fe and/or Co substantially and an aperture is 38 micrometers. It remains to the above sieve and an aperture is 500 micrometers. The manufacture approach of the sintered magnet characterized by being what passes the following sieves.
[Claim 7] The manufacture approach of the sintered magnet of claim 6 which makes the ratio of the powder of the hardener for grain boundary phases in said mixture 2 - 20 % of the weight.
[Claim 8] The manufacture approach of the sintered magnet of claims 6 or 7 that Nd occupies 50% or more of R of said hardener for grain boundary phases.
[Claim 9] The manufacture approach of one sintered magnet of claims 6-8 which manufacture said hardener for grain boundary phases with a melt quenching method.
[Claim 10] The manufacture approach of one sintered magnet of claims 6-9 which sinter at the temperature more than the melting point of said hardener for grain boundary phases.
[Claim 11] The manufacture approach of one sintered magnet of claims 6-10 which sinter in a vacuum.
[Claim 12] Consistency 5.5g/cm3 Consistency change is the above Plastic solid 0.2 g/cm3 The manufacture approach of one sintered magnet of claims 6-11 which have the process sintered so that it may become the above.
[Claim 13] Anti-chip box reinforcement is 2 0.3 kgf(s)/mm. The manufacture approach of one sintered magnet of claims 6-12 which sinter the Plastic solid which it is above.
[Claim 14] Compacting pressure is 8 t/cm2. The manufacture approach of one sintered magnet of claims 6-13 which they are above.
[Claim 15] The manufacture approach of one sintered magnet of claims 6-14 which manufacture one sintered magnet of claims 1-5.

[Translation done.]

R3

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(71)出願人　000003067
ティーディーケイ株式会社
東京都中央区日本橋１丁目13番１号

(72)発明者　福野　亮
東京都中央区日本橋一丁目13番１号　ティーディーケイ株式会社内

(72)発明者　中村　英樹
東京都中央区日本橋一丁目13番１号　ティーディーケイ株式会社内

(72)発明者　西沢　剛一
東京都中央区日本橋一丁目13番１号　ティーディーケイ株式会社内

(74)代理人　弁理士　石井　陽一

(54)【発明の名称】　焼結磁石およびその製造方法

(57)【要約】
【目的】　Ｒ－Ｔ－Ｂ系焼結磁石の製造において焼結時の寸法変化を抑えることにより焼結後の研削加工を不要として、安価な薄肉磁石を提供する。
【構成】　Ｒ（Ｒは、Ｙを含む希土類元素の少なくとも１種である）、Ｔ（Ｔは、Ｆｅ、またはＦｅおよびＣｏである）およびＢを含有し、$R_2T_{14}B$を主相とする平均粒子径２０μm 以上の粉末と、Ｒを７５～９７重量％含み、残部が実質的にＦｅおよび／またはＣｏであって、開きが３８μm 以上のフルイに残留し、開きが５００μm 以下のフルイを通過する粉末とを含む成形体を焼結して、閉空孔を３～１５体積％含む焼結磁石を製造する。

【特許請求の範囲】

【請求項１】　Ｒ（Ｒは、Ｙを含む希土類元素の少なくとも１種である）、Ｔ（Ｔは、Ｆｅ、またはＦｅおよびＣｏである）およびＢを含有する焼結磁石であって、閉空孔を３～１５体積％含むことを特徴とする焼結磁石。

【請求項２】　密度が７．１５g/cm^3 以下である請求項１の焼結磁石。

【請求項３】　閉空孔１個あたりの平均投影断面積が１０００～３００００μm^2 である請求項１または２の焼結磁石。

【請求項４】　開空孔の比率が２体積％以下である請求項１～３のいずれかの焼結磁石。

【請求項５】　Ｒを３０～４５重量％、Ｂを０．５～３．５重量％含有し、残部が実質的にＴである請求項１～４のいずれかの焼結磁石。

【請求項６】　Ｒ（Ｒは、Ｙを含む希土類元素の少なくとも１種である）、Ｔ（Ｔは、Ｆｅ、またはＦｅおよびＣｏである）およびＢを含有する焼結磁石を、主相用母合金の粉末と粒界相用母合金の粉末との混合物を成形した後、焼結することにより製造する方法であって、
前記主相用母合金が、実質的に$R_2T_{14}B$から構成される結晶粒を有し、平均粒子径が２０μm 以上であり、
前記粒界相用母合金が、Ｒを７０～９７重量％含み、残部が実質的にＦｅおよび／またはＣｏであって、開きが３８μm 以上のフルイに残留し、開きが５００μm 以下のフルイを通過するものであることを特徴とする焼結磁石の製造方法。

【請求項７】　前記混合物中における粒界相用母合金の粉末の比率を２～２０重量％とする請求項６の焼結磁石の製造方法。

【請求項８】　前記粒界相用母合金のＲの５０％以上をＮｄが占める請求項６または７の焼結磁石の製造方法。

【請求項９】　前記粒界相用母合金を液体急冷法により製造する請求項６～８のいずれかの焼結磁石の製造方法。

【請求項１０】　前記粒界相用母合金の融点以上の温度で焼結を行なう請求項６～９のいずれかの焼結磁石の製造方法。

【請求項１１】　真空中で焼結を行なう請求項６～１０のいずれかの焼結磁石の製造方法。

【請求項１２】　密度５．５g/cm^3 以上の成形体を、密度変化が０．２g/cm^3 以上となるように焼結する工程を有する請求項６～１１のいずれかの焼結磁石の製造方法。

【請求項１３】　抗折強度が０．３kgf/mm^2 以上である成形体を焼結する請求項６～１２のいずれかの焼結磁石の製造方法。

【請求項１４】　成形圧力が８t/cm^2 以上である請求項６～１３のいずれかの焼結磁石の製造方法。

【請求項１５】　請求項１～５のいずれかの焼結磁石を製造する請求項６～１４のいずれかの焼結磁石の製造方法。

【発明の詳細な説明】

【０００１】

【産業上の利用分野】本発明は、焼結時の収縮が小さい希土類焼結磁石と、その製造方法とに関する。

【０００２】

【従来の技術】高性能を有する希土類磁石としては、粉末冶金法によるＳｍ－Ｃｏ系磁石でエネルギー積３２ＭＧＯｅのものが量産されている。また、近年Ｎｄ－Ｆｅ－Ｂ磁石等のＲ－Ｔ－Ｂ系磁石（ＴはＦｅ、またはＦｅおよびＣｏ）が開発され、特開昭５９－４６００８号公報には焼結磁石が開示されている。Ｒ－Ｔ－Ｂ系磁石は、Ｓｍ－Ｃｏ系磁石に比べ原料が安価である。Ｒ－Ｔ－Ｂ系焼結磁石の製造には、従来のＳｍ－Ｃｏ系の粉末冶金プロセス（溶解→鋳造→インゴット粗粉砕→微粉砕→成形→焼結→磁石）を適用することができる。

【０００３】Ｒ－Ｔ－Ｂ系磁石では、焼結磁石の他に、磁石粉末を樹脂バインダや金属バインダで結合したボンディッド磁石も実用化されている。ボンディッド磁石は、成形の際の寸法がほぼ維持されるため、寸法精度が高く、製造後に形状加工を必要としない。しかし、工業化されているＲ－Ｔ－Ｂ系のボンディッド磁石は、単ロール法等の急冷法により製造した微細結晶からなる多結晶粒子を用いているので、磁場中成形などによる異方性化は困難である。Ｒ－Ｔ－Ｂ系焼結磁石の粉砕粉は、粉砕による歪や酸化などにより保磁力が激減しているため、ボンディッド磁石の原料粉として用いることはできない。なお、Ｒ－Ｔ－Ｂ系合金インゴットの粉砕粉を水素と反応させて、希土類水素化物とＴのほう化物とＴとに分解し、所定温度で脱水素することにより、個々の粒子内で結晶方位の揃った微細結晶を析出させる提案もなされている。この方法で得られた多結晶粒子は磁場配向が可能であり、微細結晶により高保磁力が得られるが、水素を用いるため工程が複雑となるので、実用化されていない。

【０００４】一方、Ｒ－Ｔ－Ｂ系焼結磁石では、実質的に単結晶粒子からなる粉末を磁場中で成形するため、容易に異方性磁石が得られ、しかもバインダを用いないため、高特性が得られる。しかし、焼結法では、成形体が焼結反応時に著しく収縮し、その収縮が不均一であるため、成形体の寸法精度の維持が難しい。この収縮は、成形体中の粒子の配向度や密度のばらつきなどにより異なる。異方性焼結磁石では、磁化容易軸方向とそれに垂直な方向とで収縮率が異なり、例えば、成形体の密度が４．３g/cm^3 のとき、磁化容易軸方向で２２％程度、それに垂直な方向で１５％程度となり、焼結後の密度は７．５５g/cm^3 に達する。

【０００５】異方性焼結磁石におけるこのような寸法変

化は、リング状磁石や板状磁石で薄肉のものの場合に特に問題となる。薄肉磁石において収縮率が不均一になると、反りが発生するからである。そこで、製品化に際しては、このような寸法変化を修正するために焼結体を研削加工する。しかし、研削加工には以下に述べるような問題がある。

【０００６】① 研削加工時の焼結体の材料損失量が大きくなる。例えば、厚さ１mmの薄肉板状の磁石を作製する際に１mmの反りが発生する場合、まず、厚さ３mm程度の焼結体を製造し、これの上下面を研削する必要があるので、材料の２／３が損失となる。このような損失を避けるために、厚肉の１個の母材から複数の薄肉板状磁石を厚さ１mmに切り出す場合でも、研削用カッターの歯幅が０．６mmであると約４０％もの損失が生じてしまう。また、薄肉の焼結体は機械的強度が小さいので、加工時の衝撃や取り扱いの際に欠けや割れが発生しやすく、歩留りが低くなってしまう。

【０００７】② 磁気特性が低下する。$Nd_2Fe_{14}B$系焼結磁石の保磁力は、結晶粒界のＮｄリッチ相の存在に依存していることは、様々な論文などにおいて詳しく報告されている。この系の焼結磁石を加工する際には、応力により加工面に近い領域の結晶粒界にクラック等が生じ、加工面から０．１～０．２mmの深さまでの領域で保磁力が失われてしまう。加工面近傍における磁石特性の消失は、厚肉の磁石では無視し得るものであっても薄肉磁石では影響が大きく、磁石全体としての磁気特性劣化が明白になってしまう。なお、加工により保磁力が消失した領域を酸エッチングにより除去することも可能であるが、焼結体の損失量がさらに増大し、製造コストも増加してしまう。

【０００８】このような事情から、長手方向長さ／厚さが１０以上に達する薄肉異方性磁石では、通常、Ｓｍ－Ｃｏ系ボンディッド磁石が用いられており、コスト高が問題となっている。Ｒ－Ｔ－Ｂ系の薄肉焼結磁石も存在するが、寸法調整のための加工が必須であり、しかも加工の際の材料歩留りが２０～３０％となるため、やはりコスト高となってしまっている。

【０００９】

【発明が解決しようとする課題】本発明は、Ｒ－Ｔ－Ｂ系焼結磁石の製造において焼結時の寸法変化を抑えることにより焼結後の研削加工を不要として、安価な薄肉磁石を提供することを目的とする。

【００１０】

【課題を解決するための手段】このような目的は、下記（１）～（15）の本発明により達成される。

（１）Ｒ（Ｒは、Ｙを含む希土類元素の少なくとも１種である）、Ｔ（Ｔは、Ｆｅ、またはＦｅおよびＣｏである）およびＢを含有する焼結磁石であって、閉空孔を３～１５体積%含むことを特徴とする焼結磁石。

（２）密度が７．１５g/cm^3 以下である上記（１）の焼結磁石。

（３）閉空孔１個あたりの平均投影断面積が１０００～３００００μm^2 である上記（１）または（２）の焼結磁石。

（４）開空孔の比率が２体積%以下である上記（１）～（３）のいずれかの焼結磁石。

（５）Ｒを３０～４５重量%、Ｂを０．５～３．５重量%含有し、残部が実質的にＴである上記（１）～（４）のいずれかの焼結磁石。

（６）Ｒ（Ｒは、Ｙを含む希土類元素の少なくとも１種である）、Ｔ（Ｔは、Ｆｅ、またはＦｅおよびＣｏである）およびＢを含有する焼結磁石を、主相用母合金の粉末と粒界相用母合金の粉末との混合物を成形した後、焼結することにより製造する方法であって、前記主相用母合金が、実質的に$R_2T_{14}B$から構成される結晶粒を有し、平均粒子径が２０μm 以上であり、前記粒界相用母合金が、Ｒを７０～９７重量%含み、残部が実質的にＦｅおよび／またはＣｏであって、開きが３８μm 以上のフルイに残留し、開きが５００μm 以下のフルイを通過するものであることを特徴とする焼結磁石の製造方法。

（７）前記混合物中における粒界相用母合金の粉末の比率を２～２０重量%とする上記（６）の焼結磁石の製造方法。

（８）前記粒界相用母合金のＲの５０％以上をＮｄが占める上記（６）または（７）の焼結磁石の製造方法。

（９）前記粒界相用母合金を液体急冷法により製造する上記（６）～（８）のいずれかの焼結磁石の製造方法。

（１０）前記粒界相用母合金の融点以上の温度で焼結を行なう上記（６）～（９）のいずれかの焼結磁石の製造方法。

（１１）真空中で焼結を行なう上記（６）～（10）のいずれかの焼結磁石の製造方法。

（１２）密度５．５g/cm^3 以上の成形体を、密度変化が０．２g/cm^3 以上となるように焼結する工程を有する上記（６）～（11）のいずれかの焼結磁石の製造方法。

（１３）抗折強度が０．３kgf/mm^2 以上である成形体を焼結する上記（６）～（12）のいずれかの焼結磁石の製造方法。

（１４）成形圧力が８t/cm^2 以上である上記（６）～（13）のいずれかの焼結磁石の製造方法。

（１５）上記（１）～（５）のいずれかの焼結磁石を製造する上記（６）～（14）のいずれかの焼結磁石の製造方法。

【００１１】

【作用および効果】$Nd_2Fe_{14}B$焼結磁石用の従来の成形体は、空孔がないと仮定したときの密度（理論密度：約７．６g/cm^3 ）の５５%程度の密度（約４．２g/cm^3 ）であり、４５%程度の空孔を含んでいる。そして、焼結により理論密度の９９%程度まで緻密化させるので、体積収縮率が大きくなってしまう。

【００１２】これに対し本発明では、焼結の際に、磁石内に閉空孔を所定比率で形成することにより、収縮を小さく抑える。閉空孔は磁石外部へ連通していないため、後述する従来の半焼結磁石の開放気孔（開空孔）と異なり、磁石の腐食を招くことがない。このようにして焼結の際の収縮率を小さく抑えることにより、リング状や板状の薄肉異方性磁石を製造する場合でも、形状を修正するための加工が不要となり、低コスト化および生産性向上が実現する。また、高密度成形体は抗折強度が高いので、取り扱いが容易となり、成形工程と焼結工程との間での割れや欠けの発生が少なくなる。

【００１３】本発明では、上記閉空孔を形成するために、２合金法を用いる。Ｒ－Ｔ－Ｂ系焼結磁石製造における２合金法は、組成の異なる２種の合金の粉末を混合して焼結する方法である。本発明では、２合金法において、上記主相用母合金と上記粒界相用母合金とを用いる。本発明で用いる主相用母合金の粉末は、従来の２合金法で用いるものと組成は同様であるが、粒子径は大きい。そして、本発明では、焼成時に閉空孔が形成されるように従来にない大径のＲリッチ粉末を、粒界相用母合金粉末として用いる。この粒界相用母合金粉末は、$Nd_{89}Fe_{11}$（重量比）を中心とする低融点組成を有する。粒界相用母合金の粉末は焼結時に溶融し、$R_2T_{14}B$主相に対して濡れ性の極めて良好な液相となって流動し、主相用母合金の粉末の周囲を被覆して磁石の粒界相となり、保磁力を向上させる。粒界相用母合金の粉末は大径であり、しかも溶融・流動しやすい。このため、粒界相用母合金粉末が溶融流動したあとには、焼結反応では埋まらない大きな閉空孔が残される。

【００１４】従来の２合金法でも、焼結後に粒界相となるＲリッチ粉末を添加しているが、従来のＲリッチ粉末は小径であるため、焼結体中に閉空孔は残存しない。そもそも従来の２合金法でＲリッチ粉末を添加するのは、保磁力を向上させると共に液相焼結を促進して磁石の高密度化をはかるためである。Ｒリッチ粉末を添加する２合金法において、焼結体密度を下げて収縮率を低減するという提案は従来なされてない。

【００１５】本発明の焼結磁石の表面付近には開空孔も存在するが、焼結工程の少なくとも一部を真空中または減圧雰囲気下で行なえば、液相化した粒界相用母合金が開空孔の外部への連通路を塞ぐため、開空孔の割合が減って耐食性が向上する。

【００１６】本発明では、高密度（５．５g/cm^3以上）の成形体を用い、かつ、完全に焼結させない（焼結後の密度が７．１５g/cm^3以下）ことが好ましい。これにより、焼結時の収縮率はよりいっそう小さくなる。

【００１７】本発明により製造される焼結磁石の磁気特性｛(BH)max ＝約１７～２５MGOe｝は、従来のＲ－Ｔ－Ｂ系高密度焼結磁石よりは低くなるが、Ｓｍ－Ｃｏ系のボンディッド磁石｛(BH)max ＝約１５MGOe｝よりは高くなる。Ｒ－Ｔ－Ｂ系磁石はＳｍ－Ｃｏ系磁石に比べ原料が安価である。したがって、本発明により製造される焼結磁石は、従来、薄肉磁石に用いられてきたＳｍ－Ｃｏ系ボンディッド磁石の代替品として好適である。

【００１８】なお、以下に示すように、２合金法により$R_2T_{14}B$系焼結磁石を製造する各種の提案がなされており、また、成形体を完全に焼結せずに低密度のポーラスな焼結体を製造する方法も以下に示すように知られているが、これらはいずれも本発明とは異なる。

【００１９】特開平５－４７５２８号公報には、異方性希土類ボンド磁石の製造方法が開示されている。この方法では、まず、Ｎｄ－Ｆｅ－Ｂ磁石粉末に焼結阻止剤または気化剤を混合するか、あるいは磁石粉末の表面を酸化した後、磁石粉末を磁界中において０．２～５t/cm^2の圧力で圧縮して圧粉体を作る。次いで、圧粉体を５００～１１４０℃で焼成して開放気孔を有する異方性焼成体を作り、４００～１０００℃で熱処理する。次いで、開放気孔に樹脂を含浸した後、樹脂を硬化する。同公報の表１～２には、各種焼結阻止剤を添加して７００～１０６０℃で焼成して製造した焼成体（樹脂含浸前）の密度が記載されており、これらはいずれも６．９g/cm^3以下となっている。

【００２０】同公報記載の焼結阻止剤は、酸化物、フッ化物、塩化物等、焼成中に溶融しないか、一部溶融する程度に留まるものである。同公報では、これらの焼結阻止剤が、焼成時に生じるＲリッチな液相の流動を妨げるので、高温焼成を行なっても焼成体が大きく収縮しなくなり、その結果、焼成温度を従来よりも高くでき、高い保磁力が得られるようになる、としている。同公報記載の気化剤は、カンファー、リン、硫黄、スズなどであり、これらは焼成中に気化して開放気孔を残留させる。開放気孔とは、焼成体の表面に孔の入口があり、樹脂が侵入できる大きさの連続気孔である。

【００２１】同公報の方法では６．９g/cm^3以下の低密度焼結磁石が得られているが、同公報の方法は本発明とは異なり開放気孔を形成することが目的である。同公報には、閉じた気孔が形成される前に焼成を止める旨の記載と、全空孔体積に対する開放気孔の体積の割合（有効気孔率）が高いほどよい旨の記載があり、閉空孔の比率を高くするという本発明の技術思想はみられない。同公報記載の焼結磁石は開放気孔を主体とするので、耐食性確保のために樹脂含浸が不可欠であり、しかも磁石の深奥部まで延びた開放気孔中に樹脂を到達させる必要があるので、生産性が著しく低くなってしまう。例えば、同公報の実施例では、真空引きした後に２時間樹脂含浸を行ない、さらに加圧して２時間の含浸を行なった後、樹脂の硬化処理に２時間を要している。

【００２２】本発明では閉空孔を形成するために所定組成のＲリッチ粉末を添加するので、保磁力も向上するが、同公報の方法では上記のような焼結阻止剤や気化剤

を用いて開放気孔を形成しているため、磁石中でのＲの分散が不良となり、保磁力が不十分となる。一方、保磁力向上のために磁石のＲ量を増加させると、残留磁束密度が不十分となってしまう。同公報には、焼結阻止剤の寸法に関する記載はない。なお、同公報には、保磁力向上のためにＴｂやＤｙの金属粉末を焼成体の収縮があまり大きくならない範囲で添加してもよい旨の記載がある。しかし、金属Ｔｂの融点は１３５７℃、金属Ｄｙの融点は１４０７℃であり、本発明で用いる融点の低い粒界相用母合金粉末と同様の効果は得られない。しかも、同公報には金属Ｔｂや金属Ｄｙの粒子径範囲は開示されておらず、これらを添加した実施例もない。

【００２３】また、同公報には、Ｎｄ－Ｆｅ－Ｂ合金の好ましい平均粒径は２～２０μm であると記載されており、実施例では３．５μm の微粉末を使用している。同公報には焼成前の圧粉体の密度は記載されていないが、圧粉の際に加える圧力は０．２～５t/cm^2 と低圧であり、高密度成形体は得られていないと考えられる。これらの点でも、同公報記載の方法は本発明とは異なる。

【００２４】特開昭６０－２３０９５９号公報には、Ｎｄ－Ｆｅ－Ｂ合金粉末とＮｄ－Ｃｏ合金粉末との混合物（平均粒径３～７μm ）を焼結する方法が開示されているが、同公報の実施例では密度７．４g/cm^3 の緻密な焼結磁石を作製しており、閉空孔を形成する本発明とは全く異なる。

【００２５】特開昭６３－９３８４１号公報には、Ｒ－Ｔ－Ｂ系合金粉末とＲ－Ｘ（Ｘは、Ｆｅ、またはＦｅとＢ、Ａｌ、Ｔｉ、Ｖ、Ｃｏ、Ｚｒ、Ｎｂ、Ｍｏの１種以上との混合物）合金粉末との混合物を焼結する方法が開示されている。このＲ－Ｘ合金粉末は、溶融物を急冷することにより製造され、焼結助剤としてはたらく。同公報の実施例では、１t/cm^2 で成形して１０００～１２００℃で焼結し、密度７．４３g/cm^3 の緻密な焼結磁石を製造している。同公報には、実施例において１～５００μm のＲ－Ｘ合金粉末を用いた旨の記載があるが、実施例で得られた焼結磁石は密度７．４３g/cm^3 の緻密なものである。同公報には、あえて空孔を形成することにより焼結時の収縮を抑えるという技術思想はみられない。

【００２６】特開昭６３－２７８２０８号公報には、$R_2T_{14}B$系磁石合金を粉末冶金法により製造する方法において、Ｐｒ、Ｔｂ、Ｄｙ値が３２～１００重量%の組成を有する液体急冷合金粉末または薄帯（アモルファスおよび微結晶）より得られる合金粉末を０～７０体積%含有する粉末成形体を焼結することが開示されている。この方法は、Ｒリッチ粉末を用いる２合金法であるが、同公報の実施例で用いているＲリッチ粉末は平均粒子径３～５μm の微粉末なので、焼結時に閉空孔は形成されない。

【００２７】特開平５－２１２１９号公報には、$R_2T_{14}B$相からなるＡ合金と、Ｒ、ＣｏＦｅ、Ｂを含有し、Ｒリッチ相を有するＢ合金とを混合して焼結する方法が開示されている。同公報の実施例では、両合金ともに平均粒径約５μm まで微粉砕されており、得られた焼結体はすべて密度７．４２g/cm^3 以上の緻密なものであり、本発明とは全く異なる。

【００２８】特開昭６３－１１４９３９号公報には、低融点元素（Ａｌ、Ｚｎ、Ｓｎ、Ｃｕ、Ｐｂ、Ｓ、Ｉｎ、Ｇａ、Ｇｅ、Ｔｅの少なくとも１種）または高融点元素を含むマトリックス材粉末と、$R_2T_{14}B$系磁性粉末とを混合して混合粉末を形成する混合工程と、前記混合粉末を成形して磁石化する磁石化工程とを有する複合型磁石材料の製造方法が開示されている。そして、前記磁石化工程として、混合粉末を成形して焼結する工程、または、混合粉末に熱間加圧を施して成形体を生成する熱間加圧工程が挙げられている。なお、熱間加圧前には、好ましくは予備成形を行なう。焼結温度はマトリックス材の融点よりも高く１１５０℃よりも低い温度であり、熱間加圧温度は３００～１１００℃、熱間加圧圧力は５～５０００kgf/cm^2 である。同公報では寸法歩留りを向上させることを課題としており、同公報には熱間成形法により製品の寸法歩留りを向上させることができる旨の記述がある。しかし、同公報の実施例では、焼結後または熱間加圧後の密度はすべて７．１g/cm^3 以上となっており、また、焼結前または熱間加圧前の成形体の密度の開示はない。同公報の実施例における$R_2T_{14}B$系磁性粉末の平均粒径は３～４μm と小径であり、低融点元素を含むマトリックス材粉末の粒径は最大でも２０～３０μm と小径である。同公報には、平均粒子径１００μm のＡｌをマトリックス材に用いて熱間加圧成形を行なった比較例があるが、この場合、密度７．５g/cm^3 の緻密な磁石が得られている。同公報の実施例における成形時の圧力および予備成形時の圧力は、いずれも１．５t/cm^2 以下と小さい。

【００２９】特開平３－８０５０８号公報には、ＲＦｅＢ系磁石を粉末冶金法により製造する方法において、磁石粉をプレス成形した後、４００～９００℃の温度範囲でポーラスな焼結体とし、それを溶融合金Nd_xFe_{1-x}（ｘ＝０．６５～０．８５）に一定時間浸漬する方法が開示されている。この方法は、磁場配向による熱収縮の異方性に起因する焼結後の変形を抑えることを目的とするものである。しかし、この方法は２合金法ではない。また、同公報の実施例で用いている$Nd_2Fe_{14}B$磁石粉末は約１０μm と小径であり、同公報には、成形圧力、成形体の密度、低温焼結後のポーラスな焼結体の密度は記載されていない。

【００３０】特開昭５５－１５２２４号公報には、Sm_2Co_{17}やPr_2Co_{17}等の２－１７系磁石を製造する際に、成形体を４００～９００℃で仮焼結後、液状プラスチックを含浸する方法が開示されている。この方法は、磁石の強度向上を目的としている。同公報の実施例

には、５～３０μm の粒子を成形して８００℃で焼結したときの収縮率が７％であったこと、１１５０℃で完全焼結したときの収縮率が約１２～１５％であったことが記載されている。そして、仮焼結体をエポキシ樹脂に浸漬して固化した後の密度が６．８０g/cm^3 であったことが記載されている。しかし、この方法は２合金法ではなく、磁石組成も本発明とは異なる。同公報では５～３０μm の小径粒子を用いており、また、同公報には仮焼結前の成形体の密度は開示されていない。

【００３１】特開昭６２－２８１３０７号公報には、Ｎｄ－Ｆｅ－Ｂ系合金インゴットを１０００～１１５０℃の温度範囲で溶体化処理し、溶体化処理したインゴットを２００μm 以下の粒径に粉砕し、粉砕した合金粉末の成形体を、５００～１０５０℃の温度範囲で焼鈍する方法と、焼鈍した成形体にプラスチックを含浸させて固化させる方法が開示されている。この方法において、５００～１０５０℃で焼鈍するのは、粉砕歪を除去して保磁力を向上させるためである。同公報の実施例では小径（平均粒径５μm ）の合金粉末を低圧力（２t/cm^2 ）で成形した後、焼鈍している。同公報には、成形体の密度、焼結体の密度は開示されていない。

【００３２】特開平４－３１４３０７号公報には、希土類元素、鉄およびボロンを基本成分とする合金を粉砕して磁場中成形した後、焼結して、ボンド磁石用バルク体を製造する方法が開示されている。この方法では、温度７００～１０００℃で３時間以下焼結することにより、理論密度の６０～９５％の密度をもつ半焼結合金のバルク体を製造する。半焼結合金は空孔をかなり含む組織であり、空孔は亀裂発展の核、さらには破壊の核となるため、小さな応力で容易に粉砕できる。よって破砕時の機械的歪の影響が少なくなる。同公報の実施例では、平均粒径３μm の微粉体を成形した後、半焼結してバルク体を製造している。この実施例には成形体の密度および半焼結時の収縮率は記載されていない。同公報記載の発明は、２合金法を用いておらず、半焼結合金のバルク体を粉砕してボンド磁石を製造する点で本発明と異なる。同公報の実施例における半焼結合金のバルク体の密度は５．６g/cm^3 以下であり、本発明における成形体密度と同程度である。したがって、同公報記載の半焼結合金のバルク体は空孔率が高すぎ、磁気特性および強度が不足するため、バルク磁石として使用することはできない。すなわち、粉砕およびボンド磁石化が必須である。このため、保磁力が劣化し、また、製造コストが高くなってしまう。

【００３３】また、特開平４－３１４３１５号公報には、特開平４－３１４３０７号公報記載の半焼結合金のバルク体を磁場中成形した後、成形体に樹脂を含浸させてボンド磁石を製造する方法が開示されている。この方法における磁場中成形は、半焼結合金のバルク体の粉砕と成形を兼ねるものである。同公報には、従来の焼結体の抗折強度が２．５t/cm^2 以上であるのに対し、半焼結合金のバルク体の抗折強度は１t/cm^2 未満と非常に小さく、粉砕が容易である旨が記載されている。同公報の実施例では、特開平４－３１４３０７号公報と同様に平均粒径３μm の微粉体を成形して半焼結し、密度５．２g/cm^3 以下のバルク体を製造し、さらに圧縮成形して樹脂含浸し、密度５．９～６．０g/cm^3 のボンド磁石を製造している。同公報記載の半焼結合金のバルク体は、特開平４－３１４３０７号公報記載の半焼結合金よりもさらに密度が低いため、圧縮成形および樹脂含浸を行なわずにバルク磁石として使用することは不可能である。このため、保磁力が劣化し、また、製造コストが高くなってしまう。

【００３４】上記したような従来の半焼結合金では、Sm_2Co_{17} 等の２－１７系磁石で３０μm の粒子からなる粉末が用いられている例があるが、$R_2T_{14}B$ 系磁石では平均粒径３μm 前後の小径粒子からなる磁石粉末の成形体を半焼結している。このような小径粒子からなる成形体を半焼結する場合、完全焼結を行なうときより低い温度で熱処理を施す必要があるが、低い温度領域では、保持温度の変化に対応して焼結体密度が大きく変化してしまう。すなわち、所定密度の半焼結体を製造するためには、厳密な温度管理が必要となり、製造コストが上昇してしまう。

【００３５】これに対し本発明では、まず、大径のＲリッチ粉末を用いる２合金法を利用する点で従来の方法とは異なる。また、磁石の主相となる粉末に大径のものを用いる点でも異なる。大径の主相用粉末を含む成形体中では、希土類元素リッチの液相を介した粒子移動が困難なので、焼結工程における保持温度が高温（例えば従来の完全焼結温度領域）であっても、完全焼結する前に焼結反応が進行しなくなる。このため、所定の低密度の焼結体が広い温度範囲で安定して得られることになり、焼結工程の管理が極めて容易となる。また、大径の粒子は凝集しにくいため、取り扱いが容易となり、特に成形時に金型への充填が容易となる。

【００３６】

【具体的構成】以下、本発明の具体的構成について詳細に説明する。

【００３７】<焼結磁石>本発明の焼結磁石は、Ｒ（Ｒは、Ｙを含む希土類元素の少なくとも１種である）、Ｔ（Ｔは、Ｆｅ、またはＦｅおよびＣｏである）およびＢを含有する。

【００３８】磁石組成は特に限定されないが、通常、
Ｒを３０～４５重量％、
Ｂを０．５～３．５重量％含有し、
残部が実質的にＴである
ことが好ましい。

【００３９】Ｒは、Ｙ、ランタニドおよびアクチニドであり、Ｒとしては、Ｎｄ、Ｐｒ、Ｔｂのうち少なくとも

１種、特にＮｄが好ましく、さらにＤｙを含むことが好ましい。また、Ｌａ、Ｃｅ、Ｇｄ、Ｅｒ、Ｈｏ、Ｅｕ、Ｐｍ、Ｔｍ、Ｙｂ、Ｙのうち１種以上を含んでもよい。希土類元素の原料としては、ミッシュメタル等の混合物を用いることもできる。Ｒ含有量が少なすぎると鉄に富む相が析出して高保磁力が得られなくなり、Ｒ含有量が多すぎると高残留磁束密度が得られなくなる。

【００４０】Ｂ含有量が少なすぎると高保磁力が得られなくなり、Ｂ含有量が多すぎると高残留磁束密度が得られなくなる。

【００４１】なお、Ｔ中のＣｏ量は３０重量％以下とすることが好ましい。

【００４２】保磁力を改善するために、Ａｌ、Ｃｒ、Ｍｎ、Ｍｇ、Ｓｉ、Ｃｕ、Ｃ、Ｎｂ、Ｓｎ、Ｗ、Ｖ、Ｚｒ、Ｔｉ、Ｍｏなどの元素を添加してもよいが、添加量が６重量％を超えると残留磁束密度の低下が問題となる。

【００４３】磁石中には、これらの元素の他、不可避的不純物あるいは微量添加物として、例えば炭素や酸素が含有されていてもよい。

【００４４】本発明の焼結磁石は、実質的に正方晶系の結晶構造の主相を有し、結晶粒界には、$R_2T_{14}B$よりもＲ比率の高いＲリッチ相が存在する。磁石の平均結晶粒径は、後述する主相用母合金の結晶粒径および焼結条件に応じたものとなる。

【００４５】本発明の焼結磁石は、閉空孔を含む。閉空孔とは、磁石表面に連通していない空孔である。閉空孔は磁石の３～１５体積％であり、好ましくは３～１２体積％である。閉空孔が少なすぎる磁石は、焼結時に大きく収縮しており、成形体の良好な寸法精度が維持されていない。閉空孔の多すぎる磁石は、磁石特性が不十分となり、強度も不足する。磁石中における閉空孔の合計容積率および後述する開空孔の合計容積率は、以下のようにして算出することができる。

【００４６】開空孔合計容積率Ｋ

式Ｉ　$K=(W_w-W)/V$

閉空孔合計容積率Ｈ

式II　$H=1-K-W/(V\cdot\rho)$

ただし、上記各式において、

Ｖ：サンプル形状から求めた体積、

Ｗ：サンプル重量、

W_w：サンプルを水中に浸漬し、１００Torr以下まで減圧して３０秒間保持した後、取り出し、サンプル表面の水をふき取った後のサンプル重量、

ρ：磁石の理論密度

である。

【００４７】閉空孔の形状および寸法は特に限定されないが、閉空孔１個あたりの平均投影断面積は１０００～３００００μm^2であることが好ましい。焼結初期に小さな閉空孔が形成された場合でも、焼結終了までに消滅してしまうため、閉空孔の平均投影断面積は一般に１０００μm^2未満にはなりにくい。すなわち、平均投影断面積が１０００μm^2未満の閉空孔を形成しようとすると、閉空孔が形成されずに焼結が進みすぎてしまうことになり、閉空孔の合計容積が小さくなって収縮率が小さくならない。また、閉空孔に隣接する結晶粒は保磁力が小さくなるが、磁石の密度が同じで閉空孔１個あたりの平均容積が小さい場合、閉空孔に隣接する結晶粒が多くなるので、高保磁力が得られにくい。一方、平均投影断面積が大きすぎると、磁石の強度が不十分となる。また、平均投影断面積が３００００μm^2を超える閉空孔を形成するためには巨大な粒界相用母合金を使う必要があるため、薄肉磁石では成形が困難となり、また、磁石の表面磁束が不均一となりやすい。閉空孔の断面積は、磁石断面の走査型電子顕微鏡写真を用いて測定することができる。測定に際しては、磁石を切断した後、切断面を研磨し、さらに切断面に金のスパッタ膜を形成した後、写真を撮影する。そして、磁石１個あたり任意の１００個以上の閉空孔について断面積を測定して平均値を求め、これを閉空孔１個あたりの平均投影断面積とする。

【００４８】本発明の焼結磁石の密度は、７．１５g/cm^{3}以下であることが好ましい。２００μm程度の大径の粒子を用いて高圧で成形すれば、成形体の密度を６．４g/cm^{3}程度と高くすることができるが、このような成形体では焼成の際に粒子移動が困難であるため、高温で焼成しても７．１５g/cm^{3}を超える密度とすることは困難である。逆に、小径の粒子を用いて低密度の成形体とした場合に７．１５g/cm^{3}を超える密度となるまで焼成すると、焼結が進みすぎて収縮率が大きくなってしまう。焼結磁石の密度がこの範囲であっても、磁石表面に連通する開空孔が多い場合には、磁石の耐食性が極端に低下するため好ましくない。開空孔の比率は、２体積％以下であることが好ましい。開空孔の比率は、前述した方法により求めることができる。

【００４９】本発明の焼結磁石は、以下に示す方法により製造することが好ましい。この方法では、成形工程において主相用母合金の粉末と粒界相用母合金の粉末との混合物の成形体を製造し、焼結工程において前記成形体を焼結する。

【００５０】<主相用母合金>主相用母合金の組成は、目的とする磁石組成に応じ、粒界相用母合金の組成とその混合比率とを考慮して適宜決定すればよいが、通常、

Ｒを２６～３５重量％、

Ｂを０．５～３．５重量％含有し、

残部が実質的にＴである

ことが好ましい。

【００５１】$R_2T_{14}B$系磁石では、Ｒリッチ相が液相となって流動することにより焼結反応が進行するが、本発明では、Ｒリッチの粒界相用母合金粉末を添加し、ま

た、収縮率を抑えるために焼結反応の進行を抑える必要があるので、主相用母合金のR含有量は少なくすることが好ましい。

【0052】主相用母合金は、前述した主相と前述したRリッチ相とを有する。主相用母合金の粉末の平均結晶粒径は特に限定されない。本発明では、磁場配向により異方性化するので、後述する粒子径としたときに単結晶粒子となるような結晶粒径であることが好ましいが、多結晶粒子であっても粒子内で結晶粒が配向していればよいので、平均結晶粒径は、例えば3～600μm程度の広い範囲から選択することができる。

【0053】主相用母合金の粉末の平均粒子径は、好ましくは20μm以上、より好ましくは50～350μmとする。平均粒子径が小さすぎると、前述した粒子大径化による効果が不十分となる。一方、平均粒子径が大きすぎると、薄肉の成形体中では磁場配向が困難となる。なお、主相用母合金粉末の平均粒子径は、粒子1個あたりの平均投影面積を算出し、これを円に換算したときの直径とする。粒子の投影面積の測定方法は特に限定されない。例えば、粉末の分散液を、粒子同士が重ならないようにガラス板上に塗布して写真を撮影し、この写真から粒子の投影面積を求めることができる。この他、前記塗布物を光ビームで走査して反射率変化を検出することにより、粒子の投影面積を求めることもできる。

【0054】主相用母合金の粉末の製造方法は特に限定されず、鋳造合金を水素吸蔵粉砕などにより粉末化する方法や、還元拡散法等のいずれを用いてもよく、焼結磁石を粉砕して粉末化してもよい。磁場配向により異方性化された焼結磁石を粉砕すれば、配向された小径の結晶粒からなる大径の多結晶粒子を得ることができるので、高残留磁束密度かつ高保磁力の磁石が得られる。

【0055】<粒界相用母合金>粒界相用母合金は、Rを70～97重量%、好ましくは75～92重量%含み、残部が実質的にFeおよび／またはCoである。粒界相用母合金に含まれるRとしてはNdが好ましく、R中の50%以上をNdが占めることがより好ましく、Rとして実質的にNdだけを用いることがさらに好ましい。R中のNd量が少なく、また、R量が少ないと、粒界相用母合金の融点が低くならず、閉空孔が形成されにくくなる。$Nd_{89}Fe_{11}$（重量比）共晶合金の融点は640℃、$Nd_{81}Co_{19}$（重量比）共晶合金は566℃であるが、$Dy_{88}Fe_{12}$（重量比）共晶合金の融点は890℃である。本発明で用いる粒界相用母合金は、Bを含まない。粒界相用母合金中のBは、磁石特性の向上に寄与せず、また、粒界相用母合金の融点の低下にも寄与しない。

【0056】本発明で用いる粒界相用母合金の粉末は、開きが38μm以上、好ましくは開きが53μm以上のフルイに残留し、開きが500μm以下、好ましくは開きが250μm以下のフルイを通過するものである。粒界相用母合金の粉末の粒子径が小さいと、所定の閉空孔を有する磁石が得られなくなる他、粒界相用母合金の粉末が酸化されやすくなる。粒界相用母合金の粒子径が大きくなりすぎると空孔が大きくなりすぎ、表面磁束が不均一となりやすい。また、磁石内に残留する空孔の寸法が磁石寸法に対して大きくなりすぎると十分な磁石強度が得られなくなる。

【0057】粒界相用母合金の製造方法は特に限定されないが、好ましくは液体急冷法を用いる。液体急冷法としては、合金溶湯を冷却基体に接触させて冷却する方法、例えば単ロール法、双ロール法、回転ディスク法等などが好ましく、ガスアトマイズ法を用いてもよい。合金溶湯の冷却は、窒素やAr等の非酸化性雰囲気中あるいは真空中で行なう。冷却速度が遅い場合、上記した組成の粒界相用母合金は、主としてNdとFe_2Ndとに相分離してしまう。これらの融点は1000℃以上と高く、また、Ndは極めて酸化されやすいため、閉空孔形成が難しくなる。液体急冷法により製造された粒界相用母合金は、アモルファス相または微結晶相を有する。

【0058】<粉砕工程および混合工程>主相用母合金の粉末と粒界相用母合金の粉末との混合物の製造方法は、特に限定されない。例えば、両母合金を混合した後、同時に粉砕して混合物を製造してもよく、各母合金を粉砕した後、両母合金を混合し、必要に応じてさらに微粉砕することにより混合物を製造してもよい。

【0059】混合物中における粒界相用母合金の比率は、好ましくは2～20重量%、より好ましくは3～12重量%とする。この比率が低すぎると磁石中に十分な閉空孔を形成することが難しくなり、この比率が高すぎると高特性の磁石を得ることが難しくなる。

【0060】各母合金の粉砕方法は特に限定されず、機械的粉砕法や水素吸蔵粉砕法などを適宜選択すればよく、これらを組み合わせて粉砕を行なってもよい。ただし、粒度分布の鋭い磁石粉末が得られることから、水素吸蔵粉砕を行なうことが好ましい。機械的粉砕には、鋭い粒度分布が得られることから、ジェットミル等の気流式粉砕機を用いることが好ましい。

【0061】<成形工程>成形工程では、両母合金の粉末の混合物を磁場中で成形する。このとき、成形体の密度が好ましくは5.5g/cm^{3}以上、より好ましくは6.0g/cm^{3}以上となるように成形を行なう。密度の小さい成形体では、十分な磁石特性を得ようとすると焼結時の収縮率が大きくなってしまい、焼結時の収縮率を小さくすると磁石特性が不十分となってしまう。成形体の密度の上限は特にないが、6.4g/cm^{3}を超える密度とすることは困難である。例えば、成形時に20t/cm^{2}以上の超高圧が必要になるため成形装置や金型が高価になり、また、成形体の形状が単純なものに制限されてしまう。成形体密度を向上させるためには多量の有機潤滑剤の利用も有効であるが、焼結前に有機潤滑剤を除去すること

が困難であり、磁石中の残留炭素が磁石特性を低下させてしまう。なお、成形体の密度は、マイクロメータなどにより測定した成形体の寸法から算出することができる。

【００６２】このように高い密度の成形体は、抗折強度が０．３kgf/mm^2 以上、さらには０．５kgf/cm^2 以上となるので、取り扱いが容易となり、割れや欠けの発生が少なくなる。

【００６３】成形圧力は特に限定されず、所望の密度の成形体が得られるように適宜決定すればよいが、好ましくは８t/cm^2 以上、より好ましくは１２t/cm^2 以上とする。成形時の磁場強度は、通常、１０ kOe以上、好ましくは１５ kOe以上とする。

【００６４】成形時に印加する磁界は、直流磁界であってもパルス磁界であってもよく、これらを併用してもよい。本発明は、圧力印加方向と磁界印加方向とがほぼ直交するいわゆる横磁場成形法にも、圧力印加方向と磁界印加方向とがほぼ一致するいわゆる縦磁場成形法にも適用することができる。

【００６５】<焼結工程>上記のようにして得られた成形体は、焼結されて磁石化される。

【００６６】本発明では、焼結体の密度から成形体の密度を減じた値（焼結時の密度変化量）が０．２g/cm^3 以上となるように焼結することが好ましい。焼結工程での密度変化が小さすぎる場合、焼結が不十分であり、磁石特性および機械的強度が不十分となる。収縮率を小さくするためには、密度変化量を好ましくは１．５g/cm^3 以下、より好ましくは１．２g/cm^3 以下とする。

【００６７】焼結時の各種条件に特に制限はなく、焼結時の密度変化などが所望の値となるように適宜選択すればよい。焼結時の保持温度は、粒界相用母合金の溶融温度以上であればよいが、上述したように、本発明では大径の粒界相用母合金粉末を用いることにより低密度磁石を形成するため、従来のいわゆる半焼結の場合よりも保持温度を高くすることができる。具体的には、９００～１１００℃で０．５～１０時間熱処理を施して焼結し、その後、急冷することが好ましい。なお、焼結雰囲気は、真空中またはＡｒガス等の不活性ガス雰囲気であることが好ましく、前述したように開空孔の比率を減らすことができる点で、真空中または減圧した不活性ガス雰囲気での焼結がより好ましい。なお、焼結工程の一部だけを真空または減圧雰囲気とする構成としてもよい。

【００６８】<その他>焼結後、保磁力向上のために時効処理を必要に応じて施す。

【００６９】磁石の耐食性を向上させるためには、開空孔を塞ぐことが好ましい。このためには、例えば、有機溶剤に樹脂を溶解した溶液中に磁石を浸漬した後、乾燥させる処理を施せばよい。なお、このような処理の後、樹脂の電着塗装や無電解めっき等により、通常の防食被覆を設けてもよい。

【００７０】本発明は、後述するような薄肉のリング状や板状の磁石の製造に好適であり、特に厚さが３mm以下である薄肉磁石の製造に本発明は適する。なお、磁石厚さが０．５mm未満となると、成形が困難となる傾向がある。

【００７１】<寸法偏差>本発明では、寸法偏差の極めて小さい焼結磁石が得られるので、焼結後、研削等による形状加工をせずに製品化することができる。

【００７２】すなわち、本発明によれば、平行部を有し、平行部の最大長さをその平均厚さで除した値が１０以上である薄肉焼結磁石において、平行部の厚さ偏差を１．５％以下とすることができ、１％以下とすることも容易であり、最大長さ／平均厚さが１５以上である薄肉磁石についても厚さ偏差をこのような範囲に収めることが可能である。平行部とは、対向する平行な２面で挟まれたブロックであり、平行部を有する磁石とは、例えば、板状磁石や円盤状磁石、リング状磁石である。平行部の厚さ偏差とは、平行部の厚さの最大値と最小値との差を平行部の最大長さで除した値である。平行部の厚さ偏差は、平行部の反りや厚さの不均一性の指標となる値であり、上記のような寸法比の薄肉焼結磁石の場合、反りや厚さの不均一さが大きくなるので、従来、一般に厚さ偏差が２．５％以上となっている。

【００７３】また、本発明によれば、円筒部を有し、円筒部の平均外径をその平均肉厚で除した値が１０以上である薄肉磁石において、円筒部の外径偏差および／または内径偏差を１．５％以下とすることができ、１％以下とすることも容易であり、平均外径／平均肉厚が１５以上である薄肉磁石についても外径偏差および／または内径偏差をこのような範囲に収めることが可能である。円筒部とは、外周面を有するか、外周面および内周面を有する円筒状ブロックであり、円筒部を有する磁石とは、例えばリング状磁石や円盤状磁石であるが、この場合の外径偏差および内径偏差は、外周面および内周面を有する円筒部を対象とする。円筒部の外径偏差とは、円筒部の外径の最大値と最小値との差を平均外径で除した値であり、内径偏差とは、円筒部の内径の最大値と最小値との差を平均内径で除した値である。円筒部の外径偏差および内径偏差は、円筒部の反りや歪、肉厚の不均一性の指標となる値であり、上記のような寸法比の薄肉焼結磁石の場合、反りや歪、肉厚の不均一さが大きくなるので、従来、一般に外径偏差および内径偏差が３％以上となっている。

【００７４】なお、円盤状磁石など、外周面だけを有する円筒部をもち、平均外径／平均厚さが１０以上、さらには１５以上である薄肉焼結磁石においても、円筒部の外径偏差を１．５％以下とすることができ、１％以下とすることも容易である。

【００７５】本明細書において、平行部の厚さ偏差は以下のようにして測定する。まず、被測定物を、その平行

部を構成する一方の面が定盤と接するように、定盤上に載置する。そして、平行部を構成する他方の面の定盤表面からの高さを、２０箇所で測定する。次に、前記他方の面が定盤表面と接するように、被測定物を裏返して定盤上に載置し、同様にして２０箇所で高さを測定する。測定位置は、測定対象の面をほぼ均等に２０に分割し、各領域内のほぼ中央の点とする。得られたすべての測定値から、最大値（T_{max}）と最小値（T_{min}）との差（$T_{max}-T_{min}$）を求める。この差を、前記平行部を構成する各面の長さ（長手方向長さ）のうちの最大値Ｌで除した値｛（$T_{max}-T_{min}$）／Ｌ｝を、厚さ偏差とする。互いに平行な面を２組以上有する薄肉磁石の厚さ偏差は、両主面を前記一方の面および前記他方の面としたときに大きな値となる。なお、薄肉磁石の説明における平均厚さには、上記のようにして得られたすべての測定値の平均を用いればよい。

【００７６】円筒部の外径偏差および内径偏差は以下のようにして求める。まず、円筒部の外径または内径を、円筒部の軸方向に連続して測定し、最大値と最小値とを求める。このとき、円筒部の軸方向両端部の０．１㎜の範囲の測定値は除外する。次に、前記円筒部をその軸を中心にして１５°回転させた後、同様な測定を行なう。このようにして、１５°間隔で周方向１８０°にわたって測定を合計１２回繰り返す。１２の最大値のうち最大のものをϕ_{max}、１２の最小値のうち最小のものをϕ_{min}とし、$\phi_{max}-\phi_{min}$を求める。次に、１２の最大値の平均と１２の最小値の平均との平均値ϕ_0を求め、ϕ_0を平均外径または平均内径とする。そして、｛（$\phi_{max}-\phi_{min}$）／ϕ_0｝を、外径偏差または内径偏差とする。なお、薄肉磁石の寸法比の説明における平均外径、平均内径には、上記ϕ_0を用いればよく、平均肉厚には、（平均外径－平均内径）／２を用いればよい。

【００７７】なお、寸法偏差の測定には、光学式などの非接触式の測定器を用いてもよく、接触式３次元測定器や、マイクロメータ、内周マイクロメータなどの接触式の測定器を用いてもよい。

【００７８】

【実施例】以下、本発明の具体的実施例を示し、本発明をさらに詳細に説明する。

【００７９】<実施例１>表１に示す焼結磁石サンプルを、以下に示す方法で作製した。

【００８０】まず、主相用母合金のインゴットを、鋳造により製造した。インゴットの組成を表１に示す。なお、組成の残部はＦｅである。これらの合金インゴットの平均結晶粒径は３００μmであった。各合金インゴットを、水素吸蔵・脱ガス反応による体積の膨張・収縮を利用して粗粉砕した後、ディスクミルにより粉砕し、表１に示す平均粒子径の粉末とした。なお、粉末の平均粒子径は、粉末の塗膜の光学顕微鏡写真から前述した方法により求めた。

【００８１】次に、合金溶湯をＡｒ雰囲気中で単ロール法により冷却し、表１に示す組成の粒界相用母合金を製造した。なお、表１に示す組成の残部はＦｅである。冷却ロールにはＣｕロールを用いた。粒界相用母合金は厚さ０．１５㎜の薄帯状であり、Ｘ線回折の結果、アモルファス状態であることが確認された。各粒界相用母合金をピンミルにより粉砕し、得られた合金粉末をフルイにより分級した。各粉末の分級に用いたフルイを、表１に示す。なお、表１には、粒子径の下限を規制する開きの小さいフルイを残留フルイとして、粒子径の上限を規制する開きの大きいフルイを通過フルイとして示してある。

【００８２】次いで、主相用母合金粉末と粒界相用母合金粉末とを混合した。粒界相用母合金粉末の添加量（混合物中の粒界相用母合金粉末の比率）を、表１に示す。

【００８３】各混合物を磁場中成形し、直径２０㎜、厚さ１．５㎜の円盤状成形体を得た。磁界強度は１２ kOeとし、磁化容易軸が成形体の厚さ方向となるように磁界を印加した。成形圧力および成形体密度を、表１に示す。

【００８４】次いで、各成形体を真空中で焼結した後、急冷した。焼結時の熱処理温度およびその温度に保持した時間を、表１に示す。焼結後、Ａｒ雰囲気中において６５０℃で１時間時効処理を施して、円盤状の焼結磁石サンプルとした。各焼結磁石サンプルの密度、焼結時の密度変化量、残留磁束密度（Ｂｒ）、保磁力（Ｈｃｊ）を、表１に示す。なお、ＢｒおよびＨｃｊの測定には、直径１５㎜、厚さ１０㎜の成形体を焼結して作製した磁気特性測定用サンプルを用いた。磁気特性測定用サンプルの製造条件は、成形体寸法以外は表１に示す各サンプルとそれぞれ同一とした。また、各サンプルの開空孔の合計容積率および閉空孔の合計容積率を、前述した方法により求めた。なお、磁石の理論密度を７．５５g/cm^3として計算した。結果を表１に示す。

【００８５】

【表１】

表　1

サンプル No.	主相用母合金 組成(重量%) R	B	平均粒子径 (μm)	粒界相用母合金 組成 (重量%)	通過フルイ (μm)	残留フルイ (μm)	添加量 (重量%)	成形圧力 (t/cm²)	密度(g/cm³) 成形体	変化量	磁石	熱処理条件 温度 (℃)	時間 (hr)	閉空孔 (体積%)	開空孔 (体積%)	Br (kG)	Hcj (kOe)
1 (比較)	28.5Nd	1.10	100	－	－**	－**	－**	10	5.83	0.78	6.61	1075	2	2.1**	9.8*	9.4	3
2 (比較)	28.2Nd	1.11	55	88Nd	75	－**	10	10	5.78	1.73	7.51*	1075	5	0.8**	0.0	11.0	18
3 (比較)	28.3Nd	1.13	150	100Nd**	250	53	7	10	5.95	0.50	6.45	1050	2.5	1.0**	13.5*	8.0	3
4 (比較)	30.0Nd	1.09	6*	89Nd	425	53	5	10	4.45*	3.01	7.46*	1050	3	0.5**	0.5	11.3	17
5 (比較)	32.0Nd	1.09	125	91Nd	250	38	8	10	5.94	0.15*	6.09	875	2	1.2**	17.8*	7.1	1
6	29.0Nd	1.10	93	87Nd +8Co+5Cu	180	38	7	10	5.83	0.92	6.75	1050	3	8.5	1.7	9.2	15
7	28.5Nd	1.11	180	82Nd	250	38	10	10	6.05	0.82	6.87	1025	2	8.0	1.0	9.0	15
8	29.5Nd	1.08	30	89Nd+11Co	425	38	7	10	5.73	1.06	6.99	1050	4	7.0	0.3	9.1	11
9	29.0Nd	1.13	90	86Nd +0.5Al+3Cu	180	53	4	10	5.78	0.90	6.68	1050	4	10.2	1.5	8.6	17
10	32.0Nd	1.10	150	75Nd	250	53	14	10	6.03	1.05	7.08	1050	7	5.7	0.5	9.1	12
11	27.0Nd +1.8Dy	1.05	220	89Nd	355	53	2.5	10	6.12	0.64	6.76	975	6	9.5	0.4	9.4	12
12	32.4Nd	1.10	100	89Nd	355	63	8	5*	5.20*	1.75	6.95	1040	4	5.0	2.9*	8.8	16
13	32.4Nd	1.10	100	89Nd	355	63	8	13	6.06	0.95	7.01	1040	4	6.5	0.5	9.0	14
14	32.4Nd	1.10	100	89Nd	355	63	8	10	5.91	1.05	6.96	1040	4	6.8	0.9	8.9	15
15	28.7Nd	1.13	200	80Nd+10Dy	425	90	6	10	6.15	0.67	6.82	1075	4	9.1	0.6	9.3	21
16	30.0Nd	1.08	40	95Nd	500	106	6	10	5.85	0.65	6.50	1100	4	12.5	1.3	8.7	14

**)本発明範囲を外れる値　*)好ましい範囲を外れる値

【００８６】次に、ＪＩＳ１級定盤を用いて、前述した方法により各サンプルの厚さ偏差を求めた。この結果、本発明サンプルでは、厚さ偏差が０．２～０．８％と著しく小さく、焼結時の不均一な収縮による反りが極めて少なかった。ただし、サンプルNo．１２は成形体の密度が低かったため焼結が進み、厚さ偏差が１．５％であった。厚さ１．５㎜の薄肉磁石においてこのように厚さ偏差が小さければ、研削加工による寸法修正をせずに製品化することが可能である。しかも、表１に示されるように、本発明サンプルでは十分な磁石特性が得られている。なお、厚さ偏差の算出に際しては、平行部の最大長さとして磁石の直径を用いた。

【００８７】これに対し、比較サンプルNo. ２では、残留フルイを用いず粒界相用母合金粉末の粒子径の下限を規制しなかったため、微細なＲリッチ粉末により焼結が進みすぎて閉空孔が少なくなっている。比較サンプルNo. ４では、粒子径の小さな主相用母合金の粉末を用いて形成した低密度の成形体を焼結したため、焼結が進みすぎて閉空孔が少なくなっている。比較サンプルNo. ２、４は、厚さ偏差が２．９～６．３％と大きく、焼結時の不均一な収縮により大きな反りが発生していることがわかった。厚さ偏差がこのように大きいと、製品化は不可能である。

【００８８】また、２合金法を用いなかった比較サンプルNo. １では、成形体密度が高く焼結による密度変化量が小さいため、厚さ偏差は０．９％と小さかったが、粒界相用母合金粉末を添加しなかったために、閉空孔率が低く開空孔率が高くなっており、耐食性が低い。そして、保磁力が著しく低くなっている。比較サンプルNo. ３では、成形体密度が高く焼結による密度変化量が小さいため、厚さ偏差は０．８％と小さかったが、粒界相用母合金として融点の高い金属Ｎｄを用いたため、焼結の際の溶融・流動が不十分となり、サンプルNo. １と同様に閉空孔率が低く開空孔率が高くなっており、保磁力も著しく低い。比較サンプルNo. ５では、低温で焼結したため、焼結による密度変化量が著しく小さくなり、サンプルNo. １と同様に閉空孔率が低く開空孔率が高くなっており、保磁力も著しく低い。

【００８９】次に、各サンプルを切断し、断面を研磨した後、断面に金のスパッタ膜を形成して走査型電子顕微鏡写真を撮影し、閉空孔１個あたりの平均投影断面積を求めた。サンプルNo. ７について、拡大率の異なる断面写真を図１の（ａ）および（ｂ）に示す。同図には、フレーク状の粒界相用母合金粉末の溶融・流動により形成された閉空孔が認められる。閉空孔の測定数は、各サンプルにつき１００個とした。この結果、本発明サンプルでは閉空孔の平均投影断面積が１５００～２５０００μm^2であったのに対し、比較サンプルNo. １、３および５では１００～７００μm^2、No. ２では８０μm^2、No. ４では５μm^2にすぎなかった。

【００９０】なお、密度が５．５g/cm^3以上の成形体は、０．４５kgf/mm^2以上の十分に高い抗折強度を示した。これに対し、サンプルNo. ４製造用の成形体（密度４．４５g/cm^3）では、抗折強度が０．１５kgf/mm^2と低かった。

【００９１】<実施例２>形状をリング状とした以外は実施例１のサンプルNo. ４および９とそれぞれ同様にして、焼結磁石サンプルNo. １０４および１０９を作製した。成形体密度は、サンプルNo. １０４では４．４３g/cm^3、サンプルNo. １０９では５．７６g/cm^3となり、それぞれサンプルNo. ４および９よりやや小さくなったが、焼結による密度変化量はそれぞれサンプルNo. ４および９と同じであった。成形体の寸法は、いずれも外径３０mm、内径２７mm、肉厚１．５mm、高さ７mmとし、成形の際には、磁化容易軸が径方向となるように磁界を印加した。

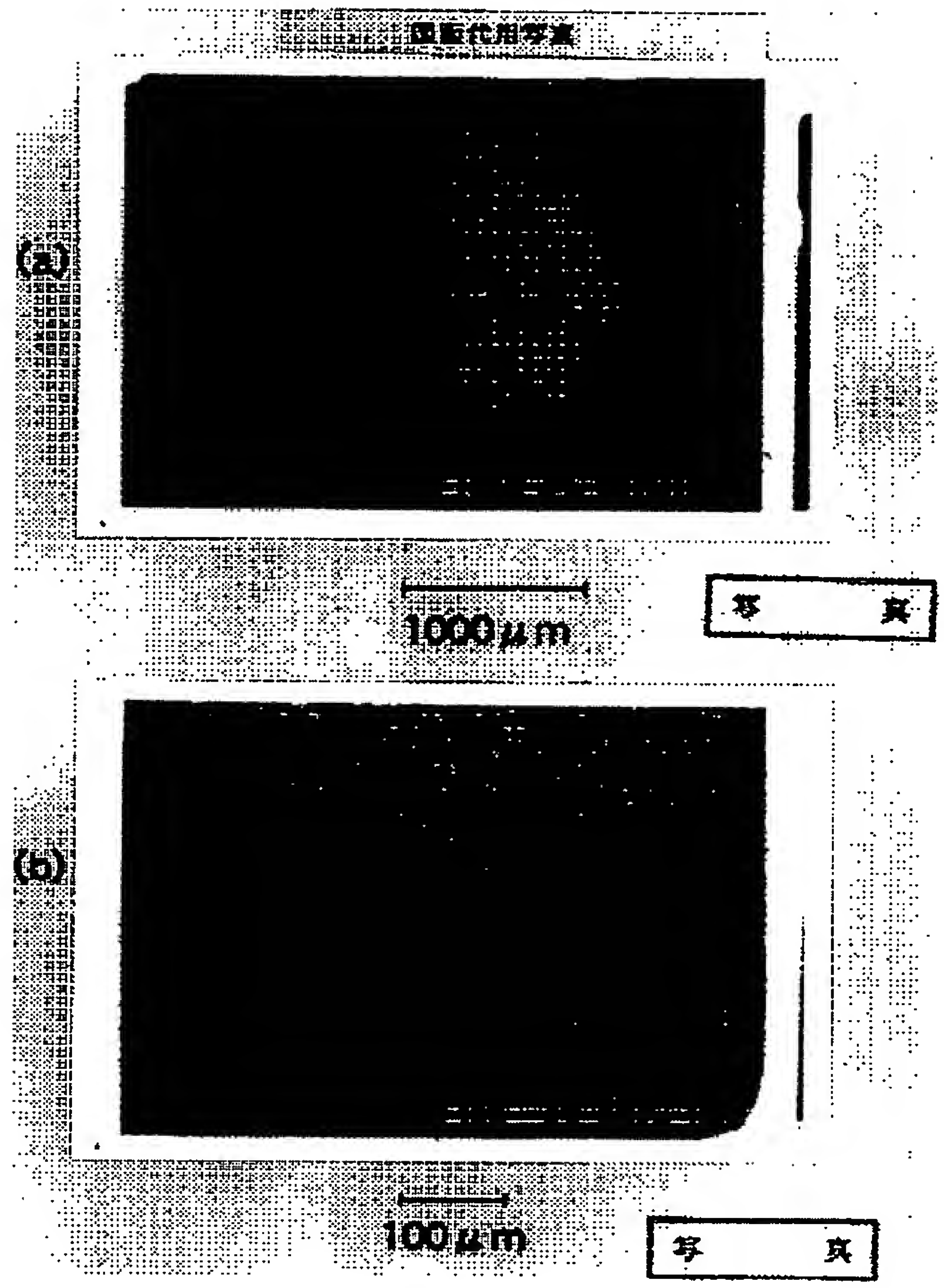
【００９２】これらのリング状焼結磁石サンプルについて、前述した方法により外径偏差および内径偏差を測定した。測定の際には各サンプルをＪＩＳ１級定盤上に外周面が接するように載置し、外径偏差は接触式３次元測定器で、内径偏差は内周マイクロメータで測定した。この結果、本発明によるサンプルNo. １０９では、外径偏差が０．３０％、内径偏差が０．３２％であり、極めて小さい値が得られたが、密度の低い成形体を焼結したサンプルNo. １０４では、外径偏差が４．５％、内径偏差が５．５％にも達し、製品化は不可能であった。

【００９３】以上の実施例の結果から、本発明の効果が明らかである。

【図面の簡単な説明】

【図１】（ａ）および（ｂ）は結晶構造を示す図面代用写真であって、本発明の焼結磁石の断面の走査型電子顕微鏡写真である。

【図１】



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